TROPICAL SCIENCE

THE QUARTERLY JOURNAL
OF THE TROPICAL PRODUCTS INSTITUTE

Volume II · Number 4

1960

LONDON
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PRICE 6s. NET

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B. G. MAEGRAITH, W. E. KERSHAW and D. DAGNALL

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EDITOR:
R. H. KIRBY, B.Com., Ph.D.

ASSISTANT EDITOR:
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This journal is successor to *Colonial Plant and Animal Products*. Any inquiries concerning articles, etc., in this journal should be addressed to The Editor, Tropical Products Institute, 56-62 Gray's Inn Road, London, W.C.1

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New Director for Tropical Products Institute: Mr. E. S. Hiscocks succeeds Dr. R. A. E. Galley

Mr. E. S. Hiscocks, M.Sc., F.R.I.C., formerly the Director of the United Kingdom Scientific Mission in Washington and Scientific Attaché at the Embassy there, has recently taken up his appointment as Director of the Tropical Products Institute in succession to Dr. R. A. E. Galley. Dr. Galley, a world authority on pesticides and Director of the Tropical Products Institute from 1953 to 1960, resigned in order to join the Shell group as Research Director and Manager of the Woodstock Agricultural Research Centre at Sittingbourne, Kent.

Mr. Hiscocks was educated at Swansea Grammar School and University College, Swansea, graduating with Honours in Chemistry in 1924. The following year he gained his M.Sc. degree and in 1935 became a Fellow of the Royal Institute of Chemistry.

From 1926 to 1939 he served in the Department of the Government Chemist (now the Laboratory of the Government Chemist) and from 1939 to 1944 was in charge of the technical branch of the Raw Materials Department, Ministry of Supply. In 1944 he became Secretary of the National Physical Laboratory and took up the appointment in the United States in 1957.

He has published several papers on physico-chemical subjects but has in more recent years made a special study of laboratory administration and in 1956 published a book under that title.

Mr. Hiscocks is married and has one son.

Developments in the Dehydration of Food

By E. G. B. Gooding and J. C. Forrest

Food Science and Atomic Energy Division,
Ministry of Agriculture, Fisheries and Food

I. General Outline

Important advances have recently been made in the techniques of food preservation by dehydration. At present, the commonest methods of drying foods to prevent spoilage due to attacks by micro-organisms are the age-old one of spreading out fish, meat, vegetables and fruit to dry in the sun and, a technique developed early in this century, the drying of food in a stream of hot air. Both methods have their advantages and limitations. The advantages are largely those of economy and simplicity; the limitations stem chiefly from the fact that, for many purposes, a dried food should be indistinguishable from the original material. In the case of sun-drying this is obviously not so; raisins, though acceptable in their own right, do not very closely resemble grapes, nor prunes plums. In the case of foods dried in hot air, these are often slow to rehydrate when put into water unless they are in very small or thin pieces. This size limitation is severe; a thickness of about $\frac{1}{4}$ in. is the most that can be used to give material that will dry satisfactorily or reconstitute completely. The lengthy exposure to heat (drying takes from six to eight hours) also tends to drive off some of the volatiles which give the characteristic flavour of many foods and air-dried foods may therefore be somewhat weaker in flavour than their counterparts.

Much research has been undertaken in the past twenty years to provide a method which would overcome these drawbacks and thus enable the full benefits of dehydration to be obtained. These benefits are very substantial; the product is stable and, under suitable conditions, keeps for long periods; the reduction of weight and volume (Plate I) results in very large transport economies; and the elimination of the need for refrigerated storage facilities also offers great savings.

Variations of the hot-air drying system include spray and roller drying, which are frequently used for liquids such as beverages.

Drying in hot air may lead to heat damage or to oxidation of the chemical constituents of foods, the result in each case being alteration of the natural flavour. Drying in vacuum might be expected to lead to improvements in each of these respects since it is carried out at lower temperatures and in the virtual absence of oxygen. On the whole, however, vacuum drying has been too costly for most foods and it has been commercially applied to relatively few. The major problem in vacuum drying has been the transfer of heat into the foodstuff. In the simple vacuum shelf dryers, the food is spread in a thin layer on trays which

rest on heated plates in a vacuum oven. Thus only the underside of the food is in contact with the heated surface and this quickly becomes dry and forms an insulating layer between the heat source and food above it. The invention in Denmark some fifteen to twenty years ago of the vacuum contact plate system went a long way to solving this problem. This involves sandwiching food between pairs of heated plates which can be brought together by the machine operator, thus permitting close thermal contact to be maintained with both sides of the food as it shrinks during dehydration. Before the technique could be developed on a commercial scale, however, experiments with this type of equipment led to the discovery of accelerated freeze drying (AFD). In this process, water contained in food in the form of ice is removed by its direct and rapid conversion into vapour without passing through the liquid phase. Although accelerated freeze drying is a new concept and the development of the technique and equipment is still only in its infancy, there is already considerable interest by industrial concerns, and plants for commercial exploitation of the process are being erected in the U.S.A., in England, and in Eire. The investigation of the vacuum contact plate system and the development of AFD were carried out by the Research Establishment and Experimental Factory of the British Ministry of Agriculture, Fisheries and Food.

The foregoing briefly summarizes the state of food dehydration today; more detailed descriptions of the techniques of hot-air drying and accelerated freeze-drying are given later.

The significance for the tropics of improvements in techniques, whether in sun-drying or by the application of the newer processes, may be considerable. For example, it has been estimated that less than 1 per cent. of the fresh fruit and vegetables grown in India is preserved but that more than 20 per cent. is wasted, mainly due to deterioration in transport. In countries where the consumer may be more than 1,000 miles from the supplier, the cost of transport is of major importance. By supplying dehydrated foods, the transport costs may be reduced by from 50 to 90 per cent. compared with those for fresh or canned foods. There would also be great packaging economies compared with canned products. These savings would, of course, be offset to a greater or lesser extent by the higher costs of the newer dehydration techniques. For the mass market it is likely that dehydrated foods produced in factories (particularly by the rather costly accelerated freeze-drying technique) would be too expensive, but scientific research could, no doubt, lead to improvements in the methods of sun-drying, with important benefits for village-scale industries. So far as the factory processes are concerned, it is possible that tropical countries could, as well as supplying the more prosperous sections of their home markets, develop useful export markets in suitable products. For example, in the meat-producing regions of sub-tropical South America, or perhaps in East Africa, accelerated freeze-drying should be able to produce excellent meat products which would have great advantages in stability and in transport to export markets. Certain tropical fruits and vegetables, too, could be preserved by this means and exported.

II. Techniques

A. *Hot-Air Drying*

The hot-air drying process is commonly used for vegetables but, with appropriate modifications in the pre-drying treatment, is also applied to fruit and, on a smaller scale, to minced meats. For the present purpose, only the dehydration of vegetables will be considered.

Raw vegetables of high initial quality are trimmed (Plate II) and cut into portions of a suitable size; the dimensions are very important as they affect both the rate of drying and the rate of reconstitution. Root vegetables are commonly cut into strips of cross-section area $\frac{3}{16} \times \frac{5}{16}$ in.; or dice generally not exceeding $\frac{1}{4}$ in. edge, while leafy vegetables are shredded (Plate III). After cutting, starchy vegetables are washed to remove surplus starch, and leafy vegetables are also washed at this stage when it is easy to remove extraneous matter.

Scalding (blanching) is the next step, the purpose being to inactivate enzymes and to effect a measure of precooking which has a beneficial effect on the texture of the finished product. If enzymes are not inactivated there is deterioration of colour and flavour during drying and subsequent storage of the product. The adequacy of enzyme inactivation is determined by testing for peroxidase activity with guaiacol-hydrogen peroxide reagent, the development of a red colour indicating the presence of active peroxidase.

Scalding is carried out in near boiling water (99°C) or, better, in steam. The addition of sulphur dioxide in the form of sodium sulphite or metabisulphite at this stage improves the colour and the storage qualities of most dehydrated vegetables. When green vegetables are scalded in water it is necessary to neutralize the organic acids liberated from the plant cells during scalding, and even to keep the liquor slightly alkaline (pH about 7·3), since in hot and acid conditions the green chlorophyll becomes altered to the dull olive-brown phaeophytin.

Leaching of solutes from plant tissues can be very severe during water scalding and, since it is the dry matter in the tissue that the dehydrator wants to recover, this can be a serious loss economically as well as nutritionally. If the leached solutes are allowed to accumulate, this leaching is to some extent reduced, but too high a concentration of solutes in the scald liquor leads to discolouration of the product and liability to browning during drying and storage. The browning which, under certain conditions, appears in dehydrated foods is the result of a complex series of chemical reactions initiated by the condensation of reducing sugars and amino acids (normal constituents of nearly all naturally occurring foodstuffs), with the formation of colourless precursors of the browning reaction. When plant extracts are heated chemical reactions of this type take place. Hence, if plant tissues are scalded in a liquor containing a high concentration of plant solutes, they are in fact being impregnated with those colourless substances that will later yield brown pigments either during drying or storage.

Scalding in steam avoids much of the leaching and all of the impregnation effect, and is greatly to be preferred for vegetables that are to be dehydrated. The duration of the scald, in steam or in water, is normally

from two and a half to three minutes. A notable exception that requires no scalding is onion; in fact, active enzymes are necessary for the full development of the onion flavour and it is undesirable to inactivate them. Plate IV shows a general view of the later stages of a vegetable preparation line.

The actual drying is usually carried out in a circuit of heated air which may be blown over trays containing the vegetables in cabinets or tunnels (Plates V and VI), or through the vegetables on a perforated conveyor belt. Since the later stages of dehydration are very slow, drying is frequently completed in deep trays on 'bins', through which heated air is blown.

In the earliest stages of hot-air drying water evaporates readily from the material and the temperature of the foodstuff therefore remains at the wet-bulb temperature of the circulating air. Rapid evaporation can thus be obtained initially using air at 210° to 220°F dry bulb, with wet bulb depression 90° to 100°F. After a short period, however, usually twenty to twenty-five minutes, the rate of drying decreases and becomes increasingly governed by the rate at which water can diffuse from the interior of the pieces of foodstuff to the evaporative surface. Consequently, the temperature of the material rises above that of the wet bulb and approaches the dry bulb temperature. It has been learned from experience that, in general, foodstuffs will suffer heat damage, usually visible as browning, if they are held at temperatures over 150°F for more than a few minutes. Consequently, it becomes necessary for the dry bulb temperature to be reduced progressively from the initial high level to no more than 150°F, or even lower in some cases.

In the later stages drying is slow, being solely governed by the rate of diffusion of water through a solid that is shrinking and becoming progressively less permeable. For most root vegetables, cut to a maximum thickness of $\frac{3}{16}$ in., it is seldom practicable to bring the final moisture content to less than 5 to 6 per cent., but for long storage life under tropical conditions lower moisture contents are desirable. Attainment of these requires thinner material, cut to not more than $\frac{1}{8}$ in. in thickness.

The operation of an air-drying system may be illustrated by considering a drying installation used for several years in the Experimental Factory at Aberdeen. This consisted of three cross draught cabinets and a deep tray dryer. The first cabinet held one batch of foodstuff, the scalded vegetable being spread on wire mesh trays at approximately $1\frac{1}{2}$ lb. per sq. ft., the trays being stacked in two trolleys, 56 trays to a trolley, giving a total batch load of about 500 lb. vegetable. The second cabinet also held one batch, the third held four batches, and each of twelve deep trays in the deep tray dryer held the partially dried vegetable from one trolley (i.e. half a batch per tray, six batches in the dryer). Drying times and temperatures were as indicated below:

1st cabinet: 210-230°F Dry bulb, 125°F Wet bulb: $\frac{1}{2}$ hour
2nd cabinet: 175-180°F Dry bulb, 120°F Wet bulb: $\frac{1}{2}$ hour
3rd cabinet: 155-160°F Dry bulb, 110°F Wet bulb: 2 hours
Fourth Stage
(Deep Tray Dryer): 145°F Dry bulb, 80°F Wet bulb: 3 hours

It will be seen that when the capacities of the four stages are taken into consideration, as well as the periods of drying at each stage, a form of semi-continuous operation can be carried out. Two trolley loads (one batch) are removed from the fourth stage; one batch is transferred from third to fourth, one from second to third, one from first to second, and one of scalded material enters the first cabinet. If this is repeated every thirty minutes a total drying time of six hours is obtained, and this was, in fact, the normal standard.

After dehydration the vegetable is packed in cans, sealed, and the atmosphere in the cans is replaced by nitrogen. The necessity for packing in hermetically sealed containers is obvious, since the uptake of water vapour must be prohibited. The additional feature of nitrogen packing is necessary because most foodstuffs contain substances which are readily oxidized by the air, and liability to oxidation increases with decrease in moisture content. Potato, for example, contains sufficient fat for strong rancid flavours to develop when the dehydrated product is stored in air; the beta-carotene of carrots becomes oxidized to various simpler compounds with marked 'off' flavours and with bleaching of the tissue; most green vegetables develop hay-like flavours and ascorbic acid is lost rapidly.

The main cause of deterioration other than oxidation is the browning reaction that has already been mentioned. This depends mainly on two factors—the temperature of storage and the moisture content of the dehydrated food.

Temperature has a profound effect. For a given product, browning is twice as rapid at 80°F as at 70°F, and about three times as rapid at 90° as at 80°F, and again three times as rapid at 100°F as at 90°F. Thus a commodity which has a storage life of two years at 70°F may remain in good condition for only one year at 80°F and six weeks at 100°F. In fact, most dehydrated foods have longer storage lives than this, especially if the moisture content is low. A vegetable with a moisture content of 2 per cent. will have about twice the storage life of a sample with 3 per cent. moisture, which in turn will have twice the resistance to browning of one with 5 per cent. moisture. Most foods with only 2 per cent. moisture will keep in good condition for a year or more at 100°F.

While it is difficult to attain moisture contents below 3 to 4 per cent. even with the most careful technique of hot-air drying, substantially lower figures can be obtained with the new accelerated freeze-drying process.

B. *Accelerated Freeze Drying*

Under appropriate conditions ice can evaporate directly to water vapour without passing through the liquid phase, i.e. by direct sublimation. The vapour pressure of pure ice at 0°C is 4·6 mm. Hg. If ice is held under a vacuum in which the surrounding partial pressure of water vapour is less, sublimation of the ice takes place. During sublimation, latent heat is absorbed and the temperature of the ice falls; with falling temperature there is a corresponding fall in the water vapour pressure of the ice. Thus the vapour pressure differential between the ice and the surrounding atmosphere diminishes and sublimation eventually ceases.

If, however, enough heat is put into the ice to maintain its vapour pressure at a level substantially above that of the surrounding atmosphere, but not enough to cause thawing, sublimation continues. This is the essential principle of freeze-drying. As with vacuum drying the transfer of heat into the foodstuff has hitherto been a major problem, and this difficulty has prevented the ordinary method of freeze drying from becoming an economic process for foods. With conventional methods of heat input, drying pieces of meat of $\frac{3}{4}$ in. thickness takes from 24 to 48 hours, and the throughput of the plant is so small that the resulting product is extremely costly. With the application of the vacuum contact plate system to freeze-drying, however, drying times of, for example, steaks of meat have been cut from approximately 24 hours to about seven hours, amply justifying the description of the process as accelerated freeze-drying. Work in Aberdeen has been aimed at providing an economically acceptable form of freeze-drying where, as described in the general outline, the product is squeezed between two heated plates in such a way that heat is supplied to both top and bottom surfaces simultaneously. This mechanism greatly increases the rate at which heat can be introduced to the material as compared with simple shelf drying and effectively reduces the drying time in the cabinet by a factor of approximately four. The factor limiting the rate of drying under these conditions is the removal of water vapour, and the design of evacuating equipment to remove the sublimed water vapour presents difficult problems. In addition, with some products of a cellular nature, the limiting factor is the rate at which water vapour can traverse the dry layer which is formed between the product surface and the ice surface.

In the accelerated freeze-drying process where heat is transferred by conduction it is advisable to have a high plate pressure on the product-heating plate interface, but since the water vapour evolved is attempting to escape from this same interface it is obvious that some stifling of the flow will occur.

The greatest single step towards the achievement of an economical freeze-drying process was the introduction of a layer of expanded metal between the product surface and the heating plate so that the heat could be transferred to the product while an adequate plate pressure was applied, and at the same time the evolved water vapour could escape to the edge of the tray and subsequently to the evacuating equipment (Plates VII and VIII). The adoption of this technique has reduced the drying time to about half that obtained during the earlier days of development of the accelerated freeze-drying process in Aberdeen and, since these initial trials indicated a reduction in drying time from classical freeze-drying on a heated shelf of about the same order, it will be appreciated that moisture can now be removed from the product very rapidly.

Consideration of the fundamental physics of the process indicates that there is no problem in applying heat to the product during the early stages of drying. However, the question of mass transfer is more complicated and we can consider the resistance to the flow of water vapour from the subliming ice surface to the evacuating equipment as having three components. Firstly, there is the resistance offered by the layer of

dried tissue formed as the ice sublimes from the product surface, with the result that the vapour must pass eventually from the ice surface to the product surface through this layer. Secondly, when the vapour escapes from the product surface it must then traverse half the width of the drying tray before escaping into the evacuated space of the cabinet. The third factor is the flow of water vapour from the edge of the drying tray to the suction port of the evacuating equipment. This flow will have a resistance decided by the design of the equipment, and if adequate cross-section is allowed and the vapour flow is kept to a reasonable level, then no particular problem arises. The second resistant force is overcome by using the expanded metal insert, the channels formed by the mesh affording an escape path for the water vapour. Although at maximum vapour flow this has a value of approximately 350 ft. per second, it has been demonstrated that the vapour pressure differential across this path is not excessive. The resistance offered by the layer of dry tissue is a function of the structure of the raw material being handled, but this can be modified in some measure by the method of pre-treatment. For example, with meat and fish and other products having a fibrous structure, it is of advantage to cut the material across the grain of the fibres before drying. This is because almost all the sublimation of water vapour takes place along the length of the grain, and if this is kept short the drying operation will be accelerated. In addition, the rate of freezing has a marked effect on the structure of such products as meat and fish steaks. With slow rates of freezing large ice crystals are formed in the tissue and when this sublimes the resulting pores provide a ready vapour escape path through the dry layer.

Before freeze-drying, the product must be completely frozen, since the method requires the removal of water vapour from the ice phase with no liquid stage drying whatever. Three rates of freezing have been studied, at $-10°C$, $-40°C$, and the even more rapid freezing by evaporation in a cabinet. The structure of meat products handled by these three methods is very different in each case. With slow freezing ($-10°C$) large ice crystals are formed, and the tissue appears to be heavily fissured when the dehydrated product is examined. This permits ready reconstitution of the product, since the water can penetrate to the centre of the piece very quickly. As the freezing rate is increased, visible damage to the tissue is reduced but, unfortunately, reconstitution time also increases.

The temperature to which the product is frozen before drying is of great importance since it is essential for complete freeze-drying that this should be below the eutectic level of the product and, with material such as fruits with high sugar contents, this freezing temperature is extremely low. The true freezing point has been studied by measuring the electrical conductivity of the flesh in the product during freezing, since there is a great difference between electrical conductivity of water in the liquid phase as compared with the solid phase. It is therefore possible to measure electrical conductivity and to state the correct freezing point with a high degree of accuracy.

Generally speaking, it is preferable to freeze the product by evaporation in the drying cabinet if possible, since this gives a much faster freezing

stage and, in addition, the fairly expensive pre-freezing equipment which would otherwise be required, can be dispensed with. However, it is not always possible to do this, since approximately 15 per cent. of the water in the foodstuff must be evaporated before the remaining water is cooled below freezing point. The initial removal of water vapour is from the liquid phase and the procedure is therefore accompanied by undesirable characteristics such as darkening of the colour, and glazing of the outer surface by migration of salts, causing denaturation of the protein. If the foodstuff is prepared in such a way that this proportion of water is available at or near the surface then the method is acceptable, but if the product is so thick that only a small proportion of water is available on the surface, then the glazed skin in the surface is also too thick. Apart from affecting the appearance of the final product, this retards freeze-drying and inhibits subsequent reconstitution. Generally speaking, freezing by evaporation is acceptable for all products which have been cut to small dimensions, i.e. dice or strips of vegetables, or minced meats and fish, and is to be recommended.

The physical considerations of the accelerated freeze-drying process were studied in a pilot plant constructed specially for the purpose (Plate IX). This equipment contains one pair of heated plates capable of holding a single tray-load of foodstuff 4 ft. square. The equipment is fully instrumented so that all physical variables can be measured and recorded during a drying cycle, and much valuable information has been obtained from its use. On the basis of experimental data gained from this experimental vacuum equipment (EVE), a specification was prepared for the Mark I AFD Cabinet which is a commercially suitable full-scale dehydration cabinet operating on the same principle (Plate X). The drying cabinet, which is capable of holding a charge of from 600-700 lb. of prepared product on 15 trays with a total area of 242 sq. ft., was manufactured by a firm in Denmark, while the four-stage steam ejector evacuating equipment was provided by a British firm. This unit was installed in Aberdeen in April 1960, and since that date a large number of proving runs have been carried out. It is evident that the plant will successfully dehydrate a wide variety of products (see table, page 198), but it appears that the 1 mm. Hg. operating pressure is rather on the high side for many fruits since their eutectic level is so low. The plant can produce a very attractive product from this material although it is dried partially from the liquid phase.

The AFD process is applicable to all foodstuffs which can be cut to greater thickness than is possible with dehydration in hot air. However, as with hot-air drying, it is imperative that raw material of optimum quality be used since in most cases it is impossible to obtain an improvement in quality simply by the dehydration process. There are two exceptions to this, namely fish and meat, where it has been shown that freeze-dried fish which has been held for a number of days on ice gives a comparable quality to dehydrated fresh raw material. In addition, during the reconstitution of freeze-dried meats it is possible to incorporate in the reconstituting water tenderizing enzymes which render originally tough meats much more palatable when they are subsequently cooked. This may

have far-reaching effects on the development of the procedure on commercial lines. The general methods of preparation are similar to those required for dehydration by hot air for vegetables and fruit with the exception that, as already mentioned, the product can be thicker than with air-drying, e.g. potatoes can be dried as half-inch square chips, fruits can be prepared as halves (plums, peaches, apricots, etc.) and whole berries such as strawberries, raspberries, blackcurrants can be effectively treated. It is also possible to dry pre-cooked prepared dishes giving a very attractive product (Plate XI). The value of AFD foods for use in unusual environments is also illustrated in Plates XII and XIII.

With meat and fish the difference between the two processes is even more pronounced, and whereas with air-drying of meats it is only possible to handle minces, giving a product of acceptable though inferior quality, with freeze-drying it is possible to dehydrate much thicker pieces. The initial stages of the preparation of the meat is to condition the carcase, bone and trim the meat. The subsequent preparation treatment varies, according to whether the product is required in the raw or cooked state.

In the case of beef, for example, the boned and trimmed joints are frozen in the blast freezer, cut to the required dimensions either on a band saw or guillotine, and then loaded on to the drying tray. The tray is then replaced in the blast freezer to reduce the product temperature to about −20°C before loading in the dehydration cabinet.

The actual drying technique is fairly standard for most products and is controlled by the temperature of the product both in the centre and on the heater-product interface. This is done by inserting thermocouples which indicate the temperature on a recorder mounted outside the cabinet. With pre-frozen material it is essential that the absolute pressure within the cabinet be reduced to the operating level as rapidly as possible and in the Mark I equipment this is a value of five minutes. The technique therefore is to load the product, establish the vacuum and the product temperature immediately reaches its lowest point due to evaporation from the surface; the heating plate temperature is then raised to its maximum, normally in the order of 120°C. At the same time, the heating plates are brought lightly into contact with the product surface, and the effect of this is to raise the product interface temperature to a fairly high level. The control of the process then depends on maintaining the temperature at this point within safe limits, and this is done by regulating the heating plate temperature to the required level. As drying proceeds the heating water is reduced in temperature until it reaches a final value of 60°C, and drying is judged to be completed when the temperature in the centre of the product also reaches this value (see figure, page 199).

By this time, the average moisture content of the product will be in the region of 1 to 2 per cent. and it has been found preferable to break the vacuum at the end of the drying cycle with commercial quality nitrogen. This is drier than atmospheric air and therefore does not give as great a moisture content increase as would be obtained by breaking the vacuum with air. Secondly, the nitrogen is preferable because the introduction of oxygen to the freeze-dried product would initiate oxidative changes.

The packaging requirements for freeze-dried foods are more stringent than for their air-dried counterparts. This is because they tend to be more fragile, but also because they have a greatly extended surface area due to their porous structure and, consequently, are much more susceptible to damage caused by moisture or oxygen uptake. This implies that, for prolonged storage, the package must be impermeable to air and water vapour and, if this were the only consideration, a tin-plate container would be the answer. However, one major advantage of these foods is the great reduction in weight which results from dehydration. There is virtually no shrinkage of the tissue and, accordingly, the bulk density of these products is low. When a tin-plate container is used the weight of the container then represents a fairly large proportion of the gross weight of the pack, and for this reason, recent tests have been aimed at developing suitable packaging materials based on aluminium foil. It is by no means certain what the final form of these packs will be, but it is apparent that no single plastic film can provide the necessary protection. Laminates of a number of plastic films give improved barrier properties, but experience has shown that the inclusion of aluminium foil has a marked effect in reducing the permeability of the laminate.

The arrangement so far used is an inner plastic film bonded to aluminium foil to permit heat sealing of the laminate, with an outer skin of cellulose acetate or similar material to give protection to the mechanically weak metal foil. This material has given good results with products such as dehydrated meat minces but there is a grave danger of pouch failure when used with products which have sharp edges such as many of the vegetables. With meat minces it has been standard practice to evacuate the pouch before sealing, and atmospheric pressure then forces the pouch into intimate contact with the contents. If there are sharp edges on the product there will be a high proportion of failures due to puncturing of the laminate. This has been overcome by using an inner cardboard liner, and it would appear to be necessary to include this feature in any commercial pack.

The danger of failure of a pack evacuated before sealing is still fairly high, since pin-holes are liable to appear at the folds in the laminate, and air will then quickly flow into the pouch. Better control of oxygen exclusion can be obtained if the air is exhausted from the pouch in a special machine, and replaced with an inert gas such as nitrogen before the pouch is sealed. This reduces the sharpness of the creases in the laminate and the possibility of holes occurring. Since the internal and external pressures are the same, the rate of ingress of oxygen through any holes which are present will be much slower.

It is extremely difficult to determine whether a gas flushed pouch has failed without carrying out an analysis of the internal atmosphere, whereas failure of a vacuum pack in a flexible laminate is immediately apparent. However, it is considered that the possibility of failure with a gas pack is so much less that this method is to be preferred.

Much work still remains to be done in this field and naturally the final package adopted will depend on the use to which the end product will be put. In situations where the weight advantage is important, it is

probable that a foil laminate will be used, but for bulk packaging, e.g. for catering purposes, it is likely that standard tin-plate containers will be more suitable.

Conclusions

The aim behind the developments in the dehydration of foods has been to produce a stable product, which when reconstituted, resembles as closely as possible the initial raw material, and which will have an adequate storage life when packed in a satisfactory container at normal ambient temperatures. There is no doubt whatever that freeze-drying gives the best results, but the improvement to be gained by this method over air-drying varies with different foodstuffs, as already mentioned. It therefore follows that the selection of method must take into account the type of foodstuff to be dehydrated and also the consumer for whom it is required.

The cost of the actual drying operation in an efficiently designed hot-air plant would be in the region of $\frac{3}{4}d.$/lb. of water evaporated as compared with about 6*d.*/lb. of ice sublimed in a freeze-drying plant of the size described. These figures take into account the depreciation and interest on capital, energy consumption, labour and maintenance attributable to the drying stage, but do not include raw material, preparation, packaging, factory overheads, etc. These latter costs will be substantially the same for both methods when producing in a similar type of pack, and consequently the ratio of actual selling costs by the two processes would not be so widely different as the costs of the drying stage suggest. It is estimated that freeze-dried foods would sell at a price about 30 to 40 per cent. higher than the corresponding air-dried material, but obviously the quality must be superior before this can be justified.

With almost all foodstuffs the superiority of the AFD foods is immediately apparent, with better colour, flavour and texture. In addition, they are more convenient to use since they absorb water more readily on reconstitution, and can be prepared for the table more quickly. However, it has been demonstrated that combinations of AFD and air-drying can be acceptable for some foodstuffs, e.g. leafy vegetables such as cabbage, leaf celery and parsley, yielding products similar in quality to their completely freeze-dried counterpart. This is obviously sound economics, since the air-drying stage greatly reduces the total cost of the dehydration operation.

In conclusion it should be pointed out that freeze-drying of a wide range of foodstuffs is already an established industry in Europe and North America and although there is only a limited throughput at present, more and more installations will be coming into production in the near future. Much thought is being given to the design of suitable equipment, and a number of manufacturers can offer drying plants with a wide range of capacities. In view of the keen competition which exists, and the scope of the potential markets which are currently being developed, it is reasonable to assume that production costs of the AFD system will be reduced. It is difficult to predict exactly how the process will develop,

PLATE I. Comparison of bulk density of cabbage in the raw state, with the same quantity dried and loosely packed in the tall can, and then compressed into two flat cans.

[facing page 194

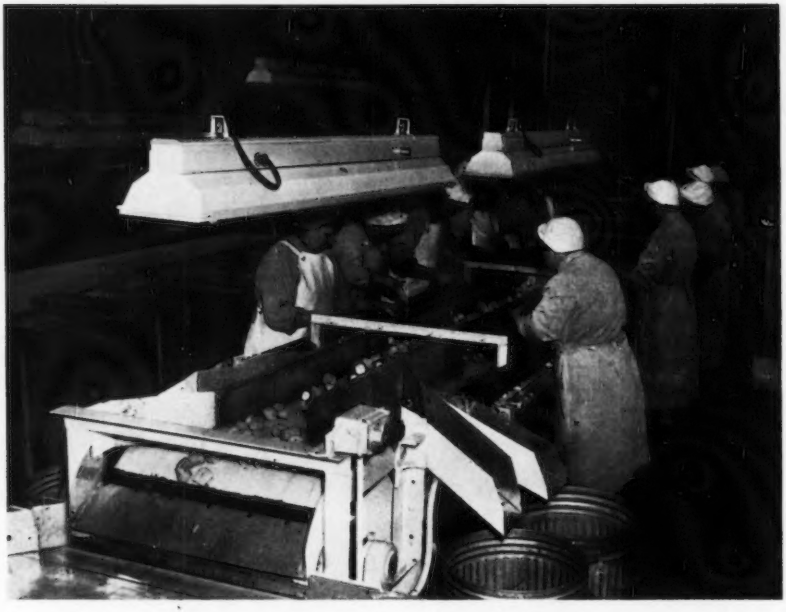


PLATE II. Trimming operation on carrot with peeled vegetable in the outside lanes and trimmed material being discharged from the centre lane

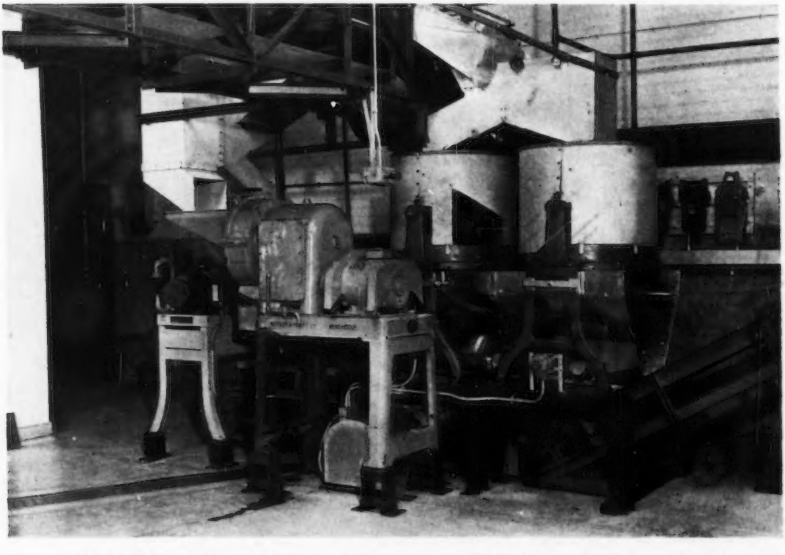


PLATE III. Vegetable cutting station in preparation line showing (*left and centre*) slicing, stripping or dicing machines for root vegetables and (*right*) two shredders for cabbage. These are fed by the overhead conveyor and discharged by the low level belts

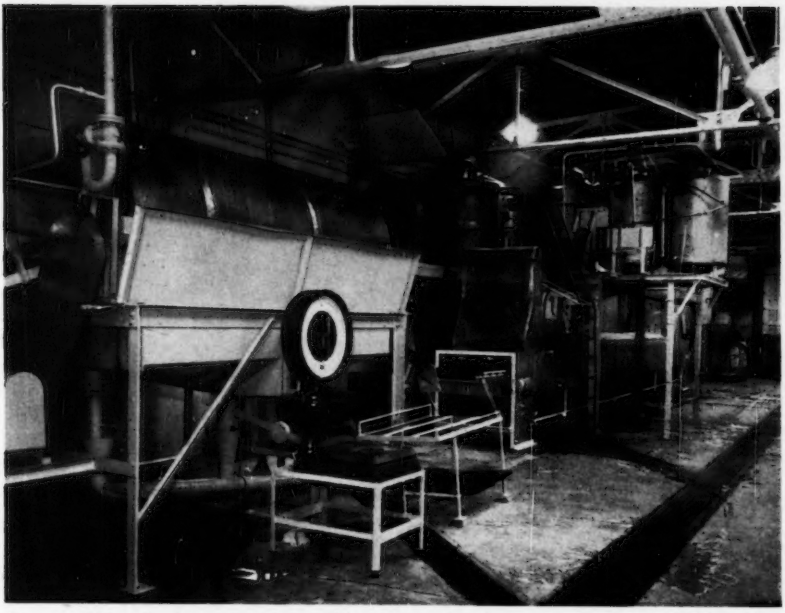


PLATE IV. Final stages of vegetable preparation line with washing machine (*left*) feeding the loading station where the product is weighed and spread on the drying trays. The trays are then fed on to the conveyor of the steam scalder (*right*), with mixing and pressurized feed tanks for dosing solutions mounted above

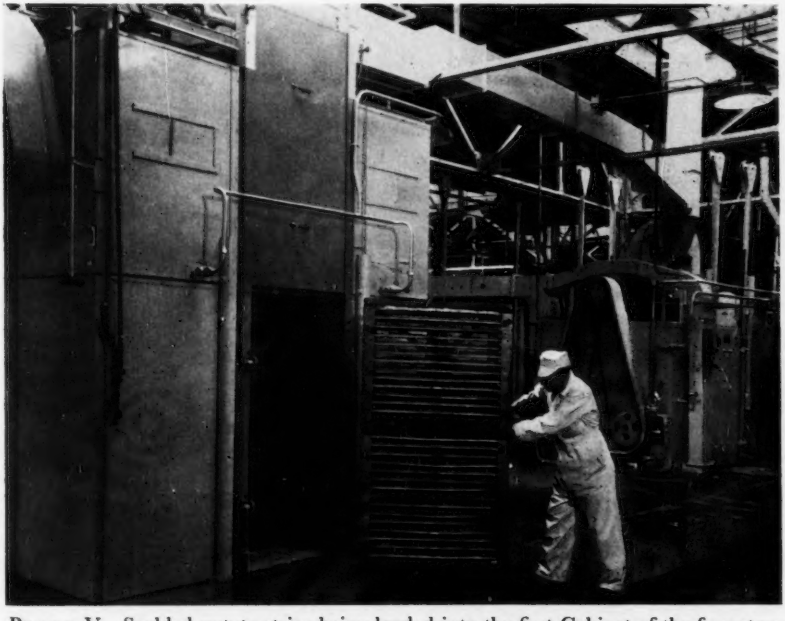


PLATE V. Scalded potato strips being loaded into the first Cabinet of the four-stage hot-air drying plant. The second cabinet is seen on the right

PLATE VI. Original three-cabinet hot-air dehydration installation. The cabinet on the left contains two trucks and others eight trucks. The second eight-truck unit was finally replaced by a deep-tray through draught drier and a new first stage cabinet was added to make the four-stage installation

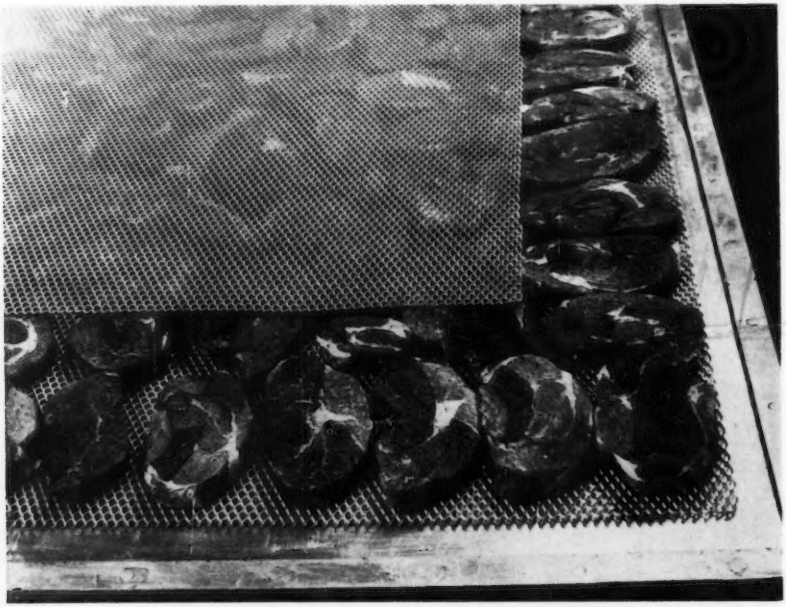


PLATE VII. Beef steaks loaded on Experimental Vacuum Equipment (EVE) drying tray with top sheet of expanded metal displaced to show the arrangement

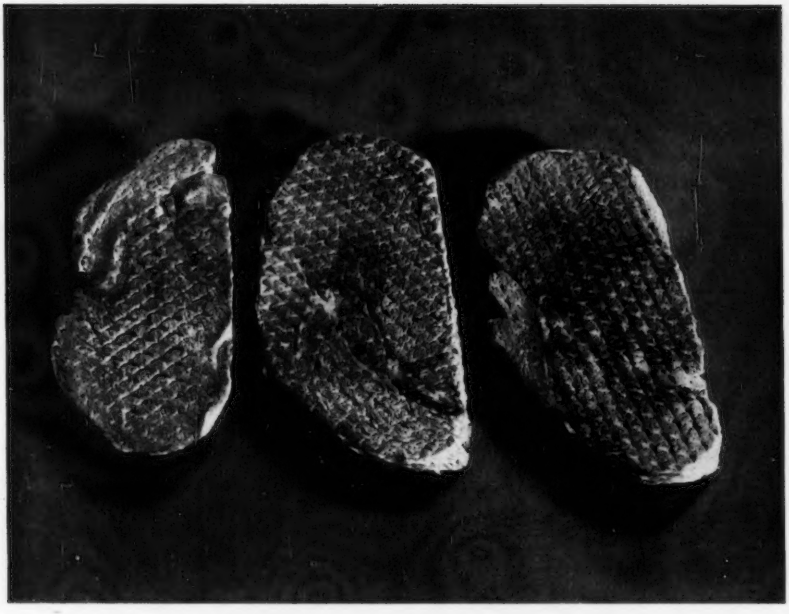


PLATE VIII. AFD beef steaks showing imprint of expanded metal inserts. This disappears completely on reconstitution

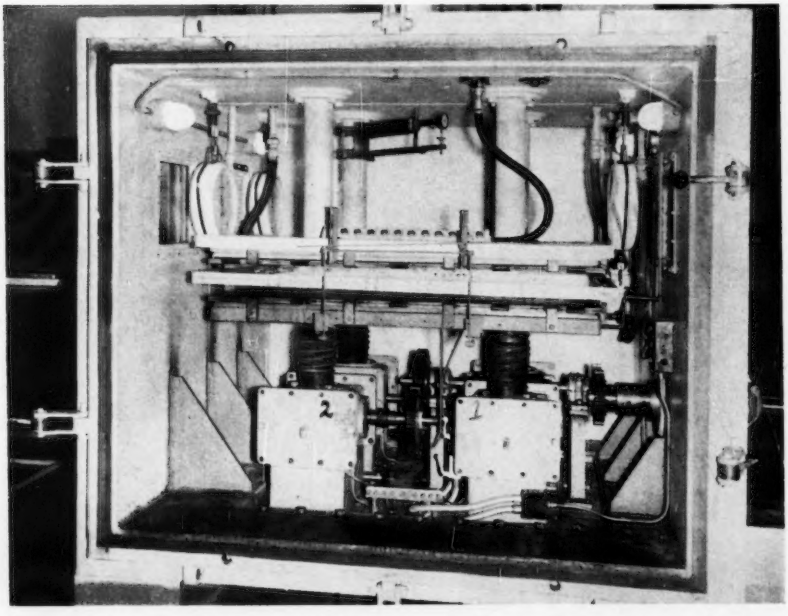


PLATE IX. Internal layout of the EVE showing the two heating plates with plate pressure jacking mechanism, and tray weighing scales opposite the viewing ports

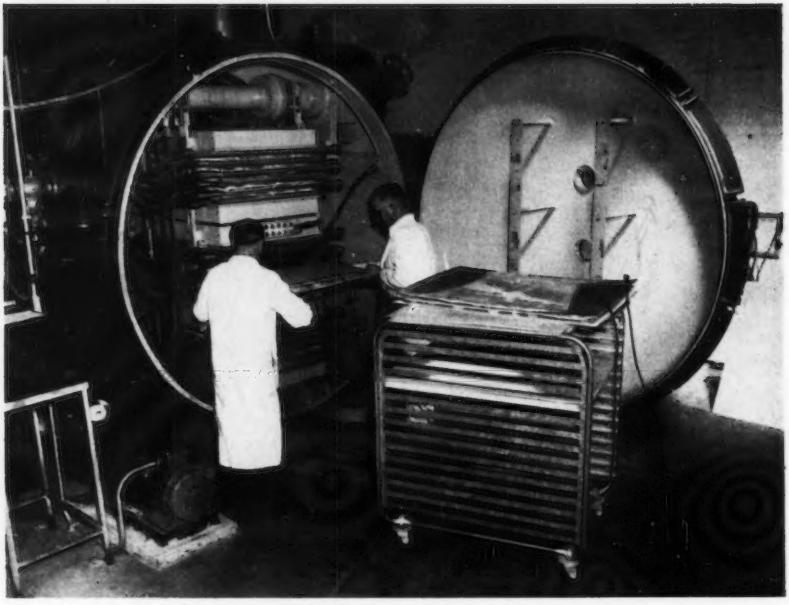


PLATE X. Mark I. Prototype AFD cabinet being loaded with pre-frozen raw cod steaks

PLATE XI. AFD meat and vegetable stew (*left*) as dried and packed and (*right*) prepared for eating

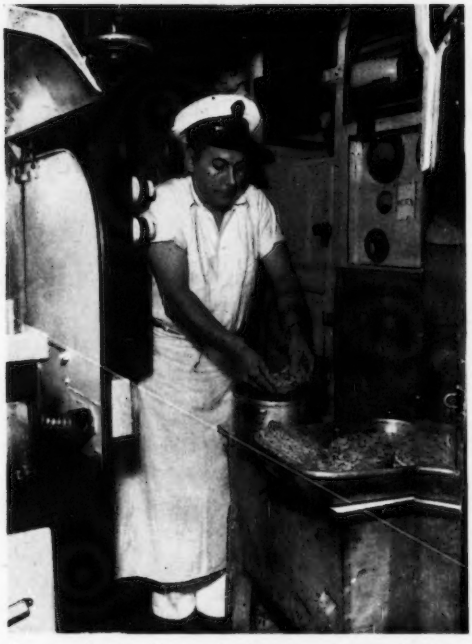


PLATE XII. The reduction in volume obtained with compressed dehydrated cabbage is of value in the confined space of a submarine galley

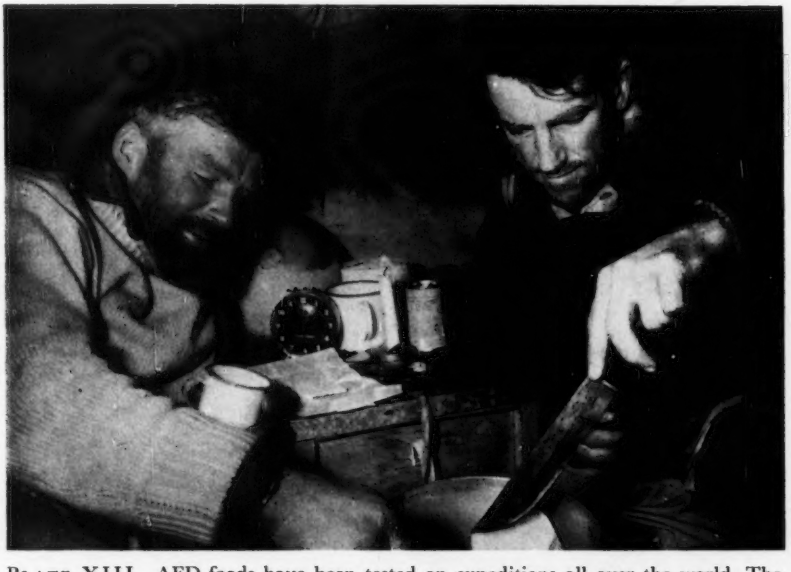


PLATE XIII. AFD foods have been tested on expeditions all over the world. The plate shows Sir Vivian Fuchs and Sir Edmund Hillary preparing AFD products in Antarctica

but it is possible that completely new products may be introduced, in some cases competing with canning or quick-freezing, but in the main augmenting the range of preserved foodstuffs which is at present available to us.

APPENDIX

Costings Summary for Dehydration Stage Only

Assume 6,000 hours operation per annum on carrot in both cases (i.e. accelerated freeze drying and hot-air drying) with a steam cost of 10*s.* per 1,000 lb. and electricity at 1¾*d.* per KW. hr. A depreciation rate of 10 per cent. is applicable, with 7 per cent. on half of the capital as an interest charge. Labour is taken at 7*s.* per hour and maintenance at 5 per cent. on the capital sum.

(1) *Accelerated Freeze Drying* (*Mark* 1 *Scale, single cabinet installation*)

Capital Costs:	£
Cabinet	9,000
Evacuating Equipment	4,550
Boiler	3,000
Installation	2,000
Total	£18,550

Running Costs:	£
Depreciation	1,855
Interest	650
Electricity (19 KW.)	830
Cooling Water (7·5 KW. for pumps)	328
Steam (1,100 lb. hr.)	3,300
Operator's wage per cabinet assuming he looks after four cabinets	525
Maintenance	928
Total	£8,416

Batch load = 500 lb.
Drying cycle = 8 hours.

$\therefore$ Annual input $= \frac{500}{8} \times 6000 = 375{,}000$ lb. prepared carrot.

$\therefore$ Cost/lb. input $= \frac{8416 \times 240}{375{,}000} = 5{\cdot}38d.$/lb. prepared carrot.

Water content of carrot = 90 per cent. average.

$\therefore$ Cost/lb. ice sublimed $= \frac{5{\cdot}38}{0{\cdot}9} = 6d./lb.$

(2) *Hot-air Drying (Multi-Cabinet Installation)*

Capital Costs:	£
Driers	6,500
Boiler	1,500
Installation	1,500
Trolleys	650
Trays (Stainless Steel)	3,750
Total	£13,900

Running Costs:	£
Depreciation	1,390
Interest	487
Electricity (24 KW.)	1,050
Steam (2,200 lb./hr.)	6,600
Operators' Wages (two men)	4,200
Maintenance	695
Total	£14,422

Throughput per annum = 6,160,000 lb. prepared carrot.

$$\therefore \text{Cost/lb. input} = \frac{14{,}422 \times 240}{6{,}160{,}000} = 0{\cdot}56d./\text{lb. prepared carrot.}$$

$$\therefore \text{Cost/lb. water evaporated} = \frac{0{\cdot}56}{0{\cdot}9} = 0{\cdot}63d./lb. \text{ prepared carrot.}$$

It should be borne in mind that the AFD plant has a much smaller throughput than the air-drying plant described, and that scaling-up of the AFD process would result in some reduction in costs.

It must be emphasised that the above refers to the *dehydration stage only*. The production of a dehydrated food involves a large number of steps, including the purchase of raw material, the preparation and processing before dehydration, packing, distribution, overheads, etc. In the United Kingdom at present, carrot dehydrated by the hot-air drying technique is sold at about 5*s.* 6*d.* per lb., equivalent to about 6·6*d.* per lb. of prepared carrot entering the dryers. On the basis of the calculation above the corresponding cost for AFD carrot would be about 11·4*d.*

Similar comparisons have been worked out for various other commodities; a few examples are given below.

Commodity	Selling price (pence per lb. of prepared material entering dryers)	
	Air dried	Freeze dried
Green Beans	15·6	21·0
Beetroot	9·0	14·9
Peas	21·3	25·8
Spinach	10·0	18·2

No similar comparison is possible for meats, as very little meat is dried by the hot-air technique and no figures are available for this method. The approximate costs of accelerated freeze-drying, drying stage only, in pence per lb. of prepared meat entering the drying cabinet, for a number of items, are as follows:

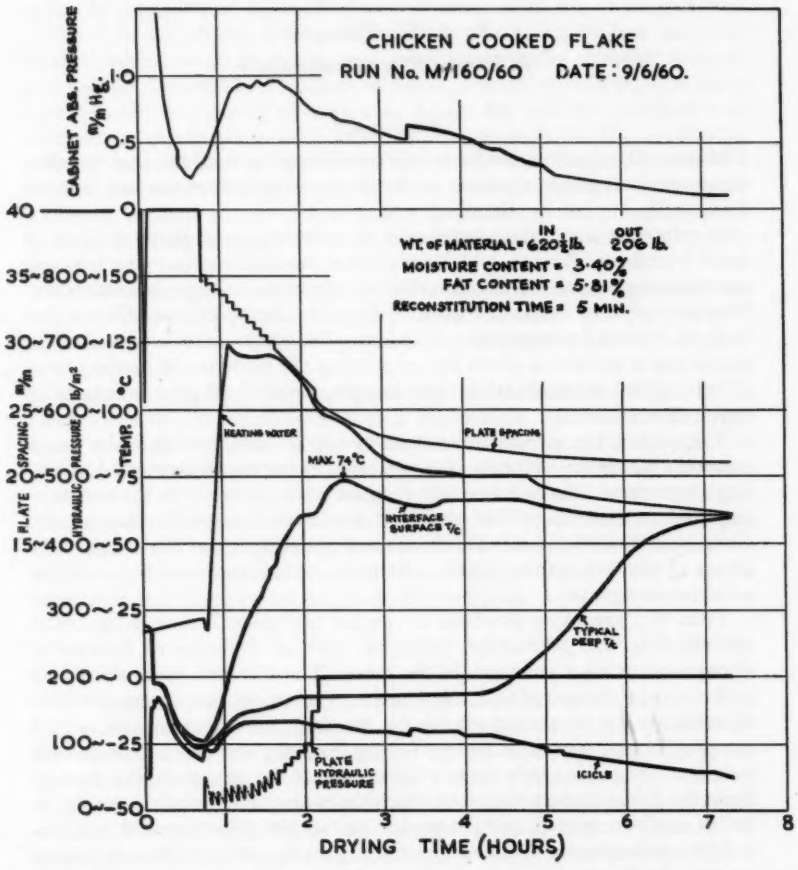
	pence
Sliced cooked beef . . .	3.0
Raw beef slices or cubes . .	3.5
Sliced cooked lamb . . .	3.0
Raw lamb slices or cubes . .	4.2
Sliced cooked pork . . .	3.8
Minced cooked meats . . .	3.2

These figures are lower than for vegetables and fruit, owing to the heavier tray loading and greater rate of throughput possible with meat, which contains a smaller proportion of water than most plant products. There seems to be considerable scope, therefore, for the AFD technique in the case of products where the water content is relatively low (approximately 50 per cent. for meat compared with about 90 per cent. for vegetables), and where initial cost of the raw material is high.

DRYING CONDITIONS FOR A RANGE OF AFD PRODUCTS IN THE MARK I CABINET

Foodstuff	Initial Weight of 15 Tray Loads *lb.*	Tray Loading *lb./sq. ft.*	Bed Depth	Drying Ratio Prepared Weight / Dried Weight	Final Moisture Content *per cent.*	Drying Time *Hours*
Cooked Pork Mince	629	2·58	25 mm.	2·23	0·7	6
Cooked Pork Mince	823	3·38	30 mm.	2·17	1·3	7½
Green Peas	480	2·0	20 mm.	4·26	1·6	9
Dipped Raw Apple Dice . . .	653	2·68	29 mm.	7·58	1·9	9½
Dipped Raw Apple Rings . . .	479	1·96	6 mm.* Treble Deck	8·96	2·0	7
Raw Beef Steaks	418	1·71	15 mm.	3·89	1·4	7
Raw Cod Steaks	750	3·14	17 mm.	5·60	1·1	7¼

* The arrangement with two intermediate layers of expanded metal gives a larger load with more uniform drying. This is because, in the case of apple, the thickness of a piece must be limited to $\frac{1}{16}$ in. to preclude internal thawing, and a single layer would probably be uneconomical.



Composite curve showing the physical conditions during the production of AFD cooked chicken flakes

An Investigation of Sampling Dried Pyrethrum Flowers from Bulk

By B. M. Church
Rothamsted Experimental Station

Summary

The general requirements for a reference sampling method, and requirements for acceptable methods of obtaining routine commercial samples from bulk, are briefly discussed.

A reference method is developed for obtaining unbiased samples of dried pyrethrum flowers, which provides a standard against which to test routine sampling and is also suitable for direct use in experimental work. The method is based on the use of 'rifflers' for the reduction of the entire bulk of flowers by repeated subdivision. The procedure is described in detail and a method is given for calculating the numbers of samples, and of laboratory determinations per sample, needed to give estimates of required accuracy.

The percentage pyrethrin content of 2-4 lb. reference samples has a standard deviation which is 1½-2 per cent. of the mean content. Laboratory errors must also be taken into account when deciding on the accuracy required in sampling. The standard deviation for spectrophotometric determinations on sub-samples from the same sample was also found to be about 1½ per cent. of the mean under favourable conditions, but could be substantially greater.

Tests of a sampling machine in routine use showed that it tended to overestimate the percentage pyrethrin content of bulks of flowers by about 0·02 or 1½-2 per cent. of the mean. The bias was fairly consistent and not large compared with the random errors of routine determinations. The bias could be accounted for by the fact that the machine, which sampled from a stream of flowers falling from the end of a conveyor belt, collected proportionately more of that part of the stream falling furthest from the belt. Random errors attributable to machine sampling are shown to be small compared with those due to sample reduction and analysis.

Some information obtained incidentally on the deterioration of ground pyrethrum flowers is summarized.

1. Introduction

Both for the orderly conduct of the pyrethrum industry, and to facilitate research work in pyrethrum agronomy and processing methods, it is important to have reliable methods of estimating the pyrethrin content of dried pyrethrum flowers. Much work has been done to develop chemical and spectrophotometric methods of analysis which will give consistent results [1–6] but, by contrast, there has been relatively little work, and no detailed results published, on the equally important problem of

obtaining representative samples of dried pyrethrum flowers from bulk for analysis.

This paper gives the results of a sampling investigation undertaken in 1960 by Rothamsted Experimental Station at the request of the East African Extract Corporation (E.A.E.C.), Nairobi, and with the support of the Department of Agriculture and Pyrethrum Board of Tanganyika.

The primary object of the investigation was to establish a sampling method which could be relied on to give intrinsically unbiased samples and enable the pyrethrin content of bulks of dried pyrethrum flowers to be estimated with specified accuracy. It was felt that the establishment of a reference sampling method would be valuable for the pyrethrum industry as a whole, both for direct use and as an ultimate standard for testing routine sampling methods. The second aim was to use this reference method to test the sampling machine currently employed by E.A.E.C., which was the basis of payments made to the Tanganyika growers.

The investigation was put in charge of the writer, who supervised the work in Nairobi, and has been forwarded at all stages by guidance and help from Dr. C. Potter, head of the Insecticides Department, Rothamsted.

So that the findings were as far as possible independent of E.A.E.C., spectrophotometric analyses of half of the samples taken in each experiment were done by the Tropical Products Institute (T.P.I.) and the writer wishes to thank Dr. A. J. Feuell and his staff for their collaboration in this work. The other half of the samples was analysed in the E.A.E.C. laboratories, Nairobi, in order to facilitate day-to-day planning of the investigation. Thanks are also due to Mr. H. Stent of the East African Industrial Research Laboratories who kindly provided working space, assistance and facilities for much of the sampling.

2. Establishment of a Reference Sampling Method

2a. *General requirements*

The fundamental requirement for a reference method of sampling from bulk is that it should be unbiased. Moreover, the lack of bias should not be dependent on the exact condition of the material sampled, or on operator characteristics which may vary from one occasion to another. A sampling method will be unbiased if every part of the bulk has an equal chance of being included in the sample. Under these circumstances, although individual samples will give values which are sometimes higher and sometimes lower than that of the bulk, the average will tend to the true value as the number of samples is increased.

It is not sufficient for the sampling procedure to be unbiased; it must also give estimates of required accuracy under all conditions likely to be encountered. Although the general order of accuracy of the sampling procedure should be known before it is used, it should also preferably include some check on its own accuracy. Provided an unbiased sampling method is available, it is easy in principle to achieve any required level of accuracy by taking repeat samples, or possibly by increasing the sample size, although this may be very laborious. However, the amount of work

involved is of secondary importance in a method which is not intended for routine use.

Consider the following simple example of an unbiased sampling procedure: suppose a bulk of 100 lb. of pyrethrum flowers is divided by any means into two roughly equal piles and that one of these piles is selected at random and weighed. Then twice the weight of the pile would be an unbiased estimate of the true weight of the bulk, because there was an equal chance that the other pile would have been selected.

If the bulk was well subdivided, so that the two piles were almost equal in weight, the estimate would be close to the true weight of the bulk. If the bulk was poorly divided and the two piles were very different, the estimate would not be close to the true weight. However, if the random choice of weighing one or other pile was repeated a large number of times, twice the average weight would approach the true weight of the bulk—not some biased value higher or lower than this—irrespective of how poorly the bulk was divided in the first case. Moreover, the number of weighings needed to estimate the bulk weight with any required accuracy could itself be estimated from the variation between the observed weights. Thus, by the expedient of making random choices, a sampling method is obtained the accuracy of which is influenced by the sample preparation but which is unbiased irrespective of that preparation.

2b. *Methods used*

The technique used in the investigation for obtaining unbiased samples was a refinement and extension of that discussed above, based on repeated sub-division of the bulk by the use of sample splitters or 'rifflers'.

The mouth of the riffler, through which the whole bulk of dried flowers was poured, was a rectangular opening 27 in. × 12 in., subdivided into twelve channels $2\frac{1}{8}$ in. wide which fed alternately into collecting bins placed to the right and the left of the riffler. The effect of pouring flowers through the riffler was to divide them into two approximately equal lots. Having done this, a random decision was taken as to which lot would be further subdivided to obtain the sample and which would be rejected. This process was repeated until the bulk was reduced to a sample of 2-4 lb. The bin used to pour the flowers through the riffler was moved gently backward and forward across the riffle mouth whilst pouring to ensure an even delivery to all chutes, and the flowers were poured slowly, so that they did not build up and block the chutes, but otherwise no special precautions were taken. Large-scale sample splitting throughout the investigation was done (under supervision) by labourers with no previous training. If the technique had been perfect, each sample would have weighed exactly $\frac{1}{2^n}$ of the total weight of the bulk after n rifflings. In practice the standard deviation was usually from 2 to 3 per cent., and never more than 5 per cent. of the mean sample weight. Deviations from the mean sample weight were not correlated with the percentage pyrethrin content of the samples.

Repeat riffling of pyrethrum flowers in quantity is laborious (in practice flowers were poured through the riffler at about 10 cwt. per hour) but

the method was chosen because it was simple and not dependent on any great skill on the part of the operator. Moreover, as about half of the flow through the riffler at any time is to one side and half to the other, it seemed unlikely that the sort of heterogeneity in the bulk which might be removed by mixing would have much effect on the sampling error. This point was important in view of the acknowledged difficulty of mixing dried flowers in bulk.

2c. *Sample reduction*

Similar methods were used for reducing the samples of dried flowers to quantities suitable for spectrophotometric analysis. Each sample was 'coarse ground' in a Wiley mill to pass a 3 mm. sieve. The ground flowers were poured twice between bowls used for handling, to eliminate any gross segregation of particles by size as they were collected from the mill. The sample was then further reduced to about ½ lb. by repeat riffling, using a smaller riffler, with 12 gaps of 0·8 in., and this quantity was fine ground in a Wiley mill to pass a 1 mm. sieve. The fine ground flowers were mixed by pouring carefully twice between metal cans and further reduced by riffling to about 50 g. A riffler with 12 gaps of 0·4 in. was used at this stage.

The reduced samples were kept in sealed tins, but the tins were usually only about half full and the samples were therefore in contact with air; the rate at which they deteriorated is discussed in Section 4.

Spectrophotometric determinations of the percentage pyrethrin content (referred to subsequently as 'pyrethrin content') were made on extract from 2 g. of ground flowers carefully weighed from the 50 g. sample, after mixing by rolling and shaking the tin. Attempts to produce segregation in the ground samples by prolonged shaking were unsuccessful. The analytical method used was that of Shukis, Cristi and Wachs[6], with detail modifications.

2d. *Laboratory errors*

Before considering the errors of sampling from bulk, the random errors introduced at the stage of laboratory extraction and spectrophotometric determination were examined. These variations due to laboratory technique, including those due to weighing of 2 g. sub-samples and all subsequent manipulations, will be referred to as 'laboratory error'.

In the course of the investigation two or more spectrophotometric determinations were made on each sample. By comparing repeat determinations, the E.A.E.C. laboratory error was estimated to be $\pm 0{\cdot}017$* (144 degrees of freedom) or 1-1½ per cent. of the mean pyrethrin content. In addition to analytical errors on a particular day, there were sometimes consistent differences between repeat determinations made on different days. These differences were partly due to sample deterioration (see Section 4) and partly to differences in the conditions under which the analytical work was done, or between analysts. In this paper such differences have been eliminated from all comparisons between samples,

*This, and all subsequent figures unless otherwise stated, are in percentage total pyrethrins.

and they have therefore also been eliminated from the above estimate of analytical error.

As the results came to hand an impression was formed that the general agreement between repeat determinations was good, but that there were occasional aberrant values. However, when all the results were examined together the analytical errors were found to be rather greater than at first supposed and close to being normally distributed, demonstrating that this stage of the process was satisfactorily under control in the E.A.E.C. laboratory:

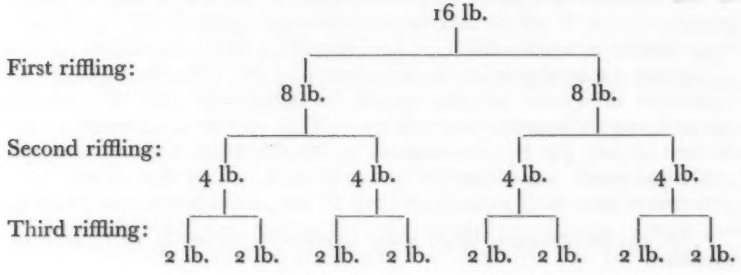
Difference between duplicate analytical determinations as a percentage of the mean	Percentage of all repeats	
	E.A.E.C. results	Normally distributed errors
Less than 1 per cent. . . .	45	41
1–2 per cent.	28	30
2–3 per cent.	18	18
3–4 per cent.	5	8
More than 4 per cent. . .	4	3
	100	100

Evidently it would be reasonable to query repeat determinations on a sample in this laboratory which differed by more than 5 per cent. of the mean.

The determination of pyrethrins on fairly large numbers of samples by spectrophotometric analysis was routine practice in the E.A.E.C. laboratories but was not normally undertaken at T.P.I. Probably because of this, the laboratory error was considerably greater at T.P.I. than at E.A.E.C. for the first series of experiments reported below, but became less as experience was gained.

2e. *The experiments*

The experiments on reference sampling may be considered in two groups. In experiments 1-5, 16 lb. batches of dry pyrethrum flowers were subdivided into eight samples, each weighing about 2 lb., by passing three times through the riffler thus:



The individual samples were coarse ground and reduced in the manner described in Section 2c to give two 50 g. samples of fine ground flowers, of which one was retained for analysis at E.A.E.C. and the other sent to T.P.I.; two or more spectrophotometric determinations were made on each sample.

The 16 lb. batches of flowers used for experiments 1 and 2 were taken from ordinary commercial consignments and each was thoroughly mixed before subdividing. For experiments 3 and 4, 16 lb. of relatively high content flowers and 16 lb. of relatively low content flowers were taken. Each of these batches was thoroughly mixed separately, by repeat riffling and bulking, and then split into two lots of about 8 lb. using the riffler. One lot from each batch was used for experiment 3, the lots being mixed together by repeat riffling and bulking before subdividing into eight samples. The other lot from each batch was used for experiment 4, but for this experiment the lots were merely put in separate layers in a bin and not mixed at all before subdivision. Experiment 5 was a repeat of experiment 4, using flowers from two other sources.

For a further series of experiments (6-13) the samples were taken from larger quantities of dried flowers. Standard bales, or equivalent quantities of unbaled flowers (about 4 cwt.), were used for these experiments. In experiments 6, 7 and 8, each bale, or equivalent, was first subdivided into 16 lots of about 28 lb. by riffling four times in the same way as in the experiments already described. Each lot was then reduced to about $3\frac{1}{2}$ lb. by riffling three times and discarding flowers falling to the left and to the right of the riffler alternately. Finally, each lot was riffled again to give sixteen pairs of samples, each weighing about 2 lb. After reducing each sample as described in Section 2c, eight of the pairs were analysed by T.P.I. and eight by E.A.E.C.

In experiments 9-13, each batch of flowers was subdivided into eight lots of about 56 lb. by riffling three times and each lot was then reduced to about $3\frac{1}{2}$ lb. by riffling a further four times and discarding from the left and right sides of the riffler alternately. After the standard sample reduction (Section 2c), four of the samples were analyzed by T.P.I. and four by E.A.E.C.

TABLE I

Characteristics of Pyrethrum from which Samples were taken

Experiment	Pyrethrins *per cent.*	Moisture *per cent.*	Percentage of Sample			
			Standing on mesh 3/16 in.	1/16 in.	1/32 in.	Through mesh 1/32 in.
6 C	1·50	8·9	—	—	—	—
7 U	1·23	9·5	71	10	10	9
8 U	1·44	11·3	83	8	5	4
9 C	1·38	12·0	80	7	6	6
10 C	1·40	9·1	69	11	8	12
11 C	1·11	13·8	93	3	2	2
12 C	1·20	10·5	77	6	8	9
13 C	1·29	11·9	80	7	6	7

C = Flowers compressed into bales which were broken for sampling.
U = Unbaled flowers.

Some important characteristics of the flowers used for experiments 6-13 are set out in Table I. The pyrethrin contents of the batches varied from 1·1 to 1·5 and moisture contents, determined by drying 5 g. samples of grist at 100-110°C. for 18 hours, varied from 9 to 14 per cent.

The extent to which the flowers had fragmented was estimated by sieve analyses. Samples of about 200 g. flowers were hand shaken for one minute on BS.410/43 sieves and the results were found to be closely reproducible on duplicate samples. Flower heads stood on the 3/16 in. mesh, broken heads and petals on the 1/16 in., while the 1/32 in. and finer fractions were rich in free achines. The proportion passing the 1/16 in. mesh varied from 4 to 20 per cent. of the total weight.

2f. *Results*

Average estimates of the laboratory variances and of the variances between samples for the series of large and small scale sampling experiments are given in Table II for the E.A.E.C. and T.P.I. determinations separately.

TABLE II

Estimates of Laboratory Variances and of Variances between Riffle Samples for Percent Pyrethrin Content ($\times 10^6$)

	Small scale experiments 1-5		4 cwt. sampling experiments 6-13	
	E.A.E.C.	T.P.I.	E.A.E.C.	T.P.I.
Laboratory variance, s_1^2 .	321 ± 77	1,223 ± 292	254 ± 44	694 ± 113
Variance between samples, $2s_2^2 + s_1^2$ . .	934 ± 223	1,779 ± 425	832 ± 151	993 ± 228
Between samples variance component, s_2^2 .	307 ± 118	278 ± 267	289 ± 79	150 ± 127

For both series of experiments the T.P.I. laboratory variance was considerably greater than that at E.A.E.C. and the T.P.I. variance between samples was correspondingly inflated.

The bottom line of the table gives estimates, s_2^2, of the variance components due to differences in pyrethrin content between samples from the same bulk, after eliminating the additional variation in the observed values due to laboratory errors. The estimates from the two laboratories are in good agreement, but are themselves subject to large errors. The standard errors of the estimates given in the table are approximate.*

The average variance of percentage pyrethrin content between independent riffle samples taken from the same bulk was about 300×10^{-6}. This corresponds to a standard deviation of $\pm 0{\cdot}017$, or about $1\frac{1}{2}$ per cent. of the mean. Although the total bulks sampled from in the large-scale experiments were almost thirty times as great as in the small-scale

*Assuming normally distributed errors, the standard error of a variance estimate, s^2, based on n observations is taken to be $\pm s^2\sqrt{\frac{2}{n-1}}$. (7)

experiments, the average variance components between samples were of much the same order for the two series. The effectiveness of the sampling is therefore not critically dependent on the size of the bulk and it is reasonable to expect similar sampling errors for even larger bulks, anyway up to a ton or so.

It is important to know whether the variability between duplicate samples is likely to be much greater when sampling from some batches of flowers than from others. In Table III estimates of the variances between samples are given for the individual experiments. The figures quoted are averages of the E.A.E.C. and T.P.I. estimates.

TABLE III

Variances of Estimates of Percent Pyrethrin Content between Riffle Samples for Individual Experiments

Experiment	Variance Estimate* $\times 10^6$	Experiment	Variance Estimate* $\times 10^6$	Experiment	Variance Estimate* $\times 10^6$
1	1241	6	1066	9	414
2	800	7	717	10	248
3	1154	8	703	11	2528
4	579			12	377
5	997			13	1278

* i.e., $(2s_2^2 + s_1^2)$

For experiments 1-5 and 9-13, estimates are each based on comparisons between eight samples and for experiments 6-8 on 16 samples

Although there are no doubt genuine differences in variability, the greater part of the differences between the estimates in this table may be attributed to chance. There is no indication that sampling errors much greater than the average are likely to be encountered under the conditions covered by these experiments. In particular, it will be noted that the variance estimates for experiments 4 and 5, for which unmixed flowers from two contrasting sources were used, were not greater than those for the other small-scale sampling experiments. It seems that little is lost by not attempting to mix flowers in bulk before taking riffle samples and no mixing was attempted for the larger-scale experiments.

The size of the individual samples ground in the Wiley mill in these experiments (2-4 lb.) was partly determined by practical convenience. From experiments 1-8 it is possible to compare estimates of variance between and within contiguous sample pairs (i.e. samples separated only by the last riffling before grinding), and these give some indication of the gain to be expected from increasing the sample size:

	Variance estimates $\times 10^6$	
	Between sample pairs	Within pairs
Experiments 1-5 .	1632 (15 d.f.)	1195 (20 d.f.)
Experiments 6-8 .	1361 (34 d.f.)	965 (34 d.f.)

Making due allowance for laboratory errors, these results suggest that if the sample size is doubled, the information on the pyrethrin content of the bulk is only increased by about one-third. Thus, if a riffle sample of 2-4 lb. does not give a sufficiently accurate estimate it will usually be best to take additional independent samples rather than increase the sample size.

2g. *Conclusions*

We have seen above that the standard deviation of the pyrethrin content of a 2-4 lb. riffle sample taken from bulk is about $\pm 0{\cdot}017$ or $1\frac{1}{2}$ per cent. of the mean. For many purposes such a sample will represent the bulk adequately, but if better representation is required, the standard error of the mean pyrethrin content of n independent samples from the same bulk is of course

$$\pm \frac{0{\cdot}017}{\sqrt{n}}.$$

The accuracy of an estimate of pyrethrin content depends not only on the sampling error but on the variability of repeat laboratory determinations on the same sample. If the pyrethrin content of a bulk of flowers is estimated by taking n independent riffle samples and doing r laboratory determinations on each, the variance of the estimate will be:

$$\frac{1}{n}\left(\sigma_2^2 + \frac{\sigma_1^2}{r}\right) \quad - - - - - - - \quad (1)$$

where σ_2^2 = variance of the actual pyrethrin content between samples

and σ_1^2 = variance between repeat determinations on the same sample (laboratory error).

The percentage of the total information provided by a sample which is obtained if r laboratory determinations are made on it is therefore:

$$100/\left(1 + \frac{\sigma_1^2}{r\,\sigma_2^2}\right) - - - - - - - - \quad (2)$$

The above experimental results show that for riffle samples of 2-4 lb. σ_2^2 is about 300×10^{-6}, and we may also take $\sigma_1^2 = 300 \times 10^{-6}$ for E.A.E.C., and $\sigma_1^2 = 1{,}000 \times 10^{-6}$ for T.P.I. The estimated percentages of the total information provided by a sample which are obtained for different numbers of laboratory determinations per sample are therefore as follows:

Number of laboratory determinations per sample		*1*	*2*	*3*	*4*	*5*	*6*	Many
Percentage of total information per sample	E.A.E.C.	50	67	75	80	83	86	100
	T.P.I.	23	38	47	55	60	64	100

If greater accuracy is required than that provided by a single laboratory determination on one sample, the number of determinations which should be made per sample depends on the relative amounts of work involved in sampling and analysis. As the analytical work is time consuming, it would rarely be worth doing more than two determinations per

sample in the E.A.E.C. laboratory, but it is always advisable to make repeat determinations on a proportion of samples to keep a good check on laboratory errors. In laboratories with larger errors it might be worth making three or four determinations per sample.

Table IV indicates the accuracy which can be expected in estimates obtained from different numbers of independent riffle samples on each of which one or two spectrophotometric determinations are made in the E.A.E.C. laboratory. Standard errors of the estimates are given at the left of the table and to the right the corresponding 5 per cent. and 2 per cent. confidence intervals are indicated.

TABLE IV

Standard Errors and Confidence Intervals for Estimates obtained from Different Numbers of Riffler Samples (E.A.E.C.)

No. of samples	Number of determinations per sample					
	1	2	1	2	1	2
	S.E. of estimated mean		5 per cent. interval (1 : 20)		2 per cent. interval (1 : 50)	
	±		±		±	
1	0·024	0·021	0·047	0·041	0·056	0·049
2	0·017	0·015	0·033	0·029	0·040	0·035
3	0·014	0·012	0·027	0·024	0·033	0·028
4	0·012	0·011	0·024	0·022	0·028	0·026
5	0·011	0·009	0·022	0·018	0·026	0·021

Thus, for example, an estimate based on one E.A.E.C. laboratory determination on a single sample will have a standard error of about $\pm 0{\cdot}024$ (or $1\frac{1}{2}$ to $2\frac{1}{2}$ per cent.); there is one chance in 20 that the difference between the estimate and the average for the bulk* will be more than 0·047 and one chance in 50 that it will be more than 0·056. Again, suppose that an estimate is required which has less than one chance in 50 of differing from the average for the bulk by more than 3 per cent. (or, say 0·04). The table shows that a single laboratory determination on each of two independent samples would satisfy this requirement.

The following general recommendations can therefore be made for unbiased reference sampling by the riffling method from bulks of dried pyrethrum flowers up to a ton or so:

(1) Decide on the number of duplicate samples (n) needed to attain the level of accuracy required, using Table IV (or equation 1 with appropriate variance estimates) as a guide. For many purposes one or two samples will be sufficient.

(2) Using a riffler with twelve channels, each about 2 in. wide, subdivide the bulk into 2^j lots by riffling j times ($2^{j-1} < n \leqq 2^j$). Select *n* of these lots at random and discard the remainder. For example if five samples are required, riffle three times dividing the bulk into eight lots; select five of these lots and discard the remaining three.

* The average for the bulk will differ from the true pyrethrin content of the bulk if the analytical method used in a particular laboratory gives biased estimates. This may be relevant when deciding how large a sampling error should be tolerated.

Whilst pouring the flowers from a bin through the riffler, the bin should be moved steadily backwards and forwards across the riffler channels to ensure an even feed of flowers to all channels; the rate of pouring should not be great enough to cause overflowing or blockage of individual channels.

(3) Reduce each lot by repeat riffling, continuing until it has been reduced to a sample of 2-4 lb. When the lot has been halved by the first riffling, make a random decision (e.g. by tossing a coin) whether to accept the portion to the left or to the right of the riffler and discard the other portion. In subsequent rifflings accept from the left and right sides alternately.

(4) Grind each sample in a Wiley mill to pass a 3 mm. sieve. Pour the sample twice between suitable receptacles to eliminate any gross segregation by particle size as the sample is collected from the mill.

(5) Using a riffler with twelve channels each about 0·8 in. wide, reduce the sample to about ½ lb. by riffling twice and accepting alternately from the right and left of the riffler.

(6) Grind the ½ lb. sample in a Wiley mill to pass 1 mm. sieve. Mix the ground sample as in (4).

(7) Using a riffler with twelve channels each about 0·4 in. wide, reduce the sample to about 50 g. by riffling four or five times and accepting alternately from the right and left of the riffler.

(8) Proceed to analysis. The relative amounts of information about the bulk obtained from different numbers of spectrophotometric determinations per sample may be estimated by using equation 2.

This method is not intended for sampling very large bulks, which are not commonly encountered in experimental work. It should be possible to test any acceptable method for sampling large bulks against this method on lots of a ton or less, because a method suitable for sampling from, e.g., 25 tons should be shown to give representative samples from any batches of 1 ton or less into which the bulk might be subdivided.

3. Routine Sampling from Bulk

3a. *General requirements*

The requirement that all portions of a large bulk of material should have a fair chance of being included in a sample is only likely to be satisfied in practice when the material is being conveyed—along a belt, through a pipe, or falling from the end of a conveyor belt or through a chute. If the material in a cross-section of the flow is always reasonably homogeneous (as might be, for example, a finely ground powder) some form of continuous flow sampling, designed to remove a fixed proportion of the flow past a certain point, may be effective. This type of sampling has the advantage of ensuring that both short- and long-term changes in the quality of the flow are reflected in the sample.

When a cross-section of the flow is likely to be heterogeneous (as is a stream of partly broken pyrethrum flowers) there may be considerable difficulty in ensuring that a continuous flow sample gives a fair representation of all types of particles. Under these circumstances, a sampling method which aims to remove the entire cross-section of material during

specified time intervals may be preferred. If the sampling intervals are sufficiently frequent, this kind of method will also give samples reflecting all except very short-term changes in the quality of flow. The samples will be unbiased, provided the entire cross-section of the flow is collected throughout the sampling intervals, and that these intervals are located at random (or at fixed intervals with an effectively random start) throughout the entire period of flow.

The most important requirement for a routine sampling method is that it should be capable of being completely specified and not subject to uncontrolled variations. For this reason mechanical methods of sampling are generally to be preferred to what may be termed 'hand sampling'. The validity of most hand sampling procedures depends not only on how well the sampling programme has been drawn up but on how well the man taking the sample interprets his instructions. If the sampler operates in a slipshod fashion the results obtained may well be useless or misleading. This personal element in sampling constitutes a serious danger, particularly for routine sampling which is usually tedious and for which it may be impracticable to employ high-grade staff. By contrast, the behaviour of a well-constructed machine will be consistent and if its performance is tested periodically and found to be satisfactory one can infer that it has taken representative samples in the intervening period.

One can nearly always think of reasons why mechanical samplers may give biased samples, and they are therefore unsuitable to provide an absolute standard and need to be tested against an established reference method. When there are theoretical possibilities of bias, some bias almost certainly exists but this may not be important in practice if the bias is very small, or so consistent that it can be allowed for. Apart from questions of bias, the extent of the random variations to be expected from sample to sample about the mean should be known for any routine sampling method and should be within required limits of accuracy.

3b. *The E.A.E.C. sampling machine*

The entire supply of flowers at E.A.E.C. taken from sacks or from broken-down bales, is passed over a conveyor belt on its way to the extraction plant. The stream falling off the end of this belt is sampled by a scoop every 55 seconds. The sampling scoop is mounted on a pair of arms keyed to a shaft which is rotated through a half-circle by a small pinion wheel driven by a toothed rack actuated by an electric thruster. The sampling action is completely automatic, a ratchet driven from the drum of the belt conveyor operating a mercury switch every 55 seconds.

The scoop samples the whole width of the falling stream. It moves into the stream from above and beyond the end of the belt until it is checked by a shock absorber in a horizontal position where it is collecting virtually the whole cross-section of the stream. In this position the scoop touches a cut-off plate which ensures that any portion of the stream falling too close to the belt to be sampled is collected and returned by hand to the belt; this portion is in fact very small—less than 0·1 per cent. of the total. A heavy counter-weight returns the scoop to its original position, emptying the contents into a collecting bin, where the individual sample grabs are bulked.

Two sampling machines of this type are in use at E.A.E.C. One (M_1) is normally used for sampling individual growers' consignments and the other (M_2) for sampling larger bulks of about 27 tons (either Northern, Southern Highlands or Kenya flowers) as they go forward for extraction. Pyrethrum flowers are fed direct from a hopper on to the belt of M_1. The steady flow of the larger quantities handled on M_2 is controlled by a 'bucket' feed: segments on the periphery of a drum are filled from a hopper and their contents are emptied on to the belt as the drum rotates. After passing under a regulating gate, an even loading of the belt results as long as the hopper is kept filled. The loading during the investigation was about 2½ oz. per foot, or 16 cwt. per hour. Each grab of a scoop collected 2-3 oz. of flowers, corresponding to an effective sampling time of about ⅓ second. The sampling time is kept short so that samples can be taken frequently from the flow, giving a good representation of all parts of the bulk, without making the total size of the sample too great. Movement of the scoop into and out of the falling stream is very rapid. The aggregate sample taken from a 27-ton consignment by M_2 amounts to some 350 lb. of flowers (a sampling fraction of about 1/200); the machine M_1 was adjusted to sample a somewhat larger fraction.

Although the sampling machines collect from the entire cross-section of the falling stream, the scoops must obviously sample selectively as they move into and out of the stream, having a relatively longer period during which they can collect from that part of the stream falling furthest from the belt. The practical importance of this possible source of bias depends on the difference between the pyrethrin content of different parts of the cross-section of the falling stream and on the proportion of the total sampling time when the machine is sampling selectively.

It is not difficult to think of other possible causes of bias. For example, the rapid movement of the scoop in and out of the falling stream, designed to minimize the selective sampling of different parts of the stream discussed above, may create eddies, or otherwise push aside part of the material which should be sampled. A further possibility suggested was that the sampling on M_2 might be so timed as to always coincide with 'peaks' or 'troughs' of the material placed on the belt from the segments of the belt feed. This possibility can be ruled out because the position of the crank which drives the feed, at the moment of sampling, moves round slowly relative to a fixed point, 14 samples corresponding to 491 complete revolutions of the crank. A detailed investigation of all possible sources of bias individually would have been a very large undertaking and the work discussed below was designed primarily to test the overall performance of the sampling machines under the conditions normally encountered.

The normal E.A.E.C. practice is to reduce the machine samples to a quantity suitable for handling in the laboratory by use of a special quartering machine. This machine consists of a slowly revolving drum of which the circumference is divided into segments. A lever keyed to the spindle on which the drum revolves, and turning at the same speed, operates a flap at every fourth segment, diverting the contents into a separate chute from that which removes the contents of the other three segments. The divisions between the segments are wide enough to ensure

that the contents of adjacent segments cannot mix due to slow operation of the spring-loaded flap. The quarter of the original sample obtained in this way is further reduced by passing it again through the quartering machine. There is every reason to think that such a method of sample reduction is unbiased and reasonably efficient, but it was not tested in the present investigation.

3c. *Tests of the sampling machines for bias*

Before the flowers used for experiments 6-13 (see Section 2e) were riffled, each batch was passed separately over the conveyor belt of a sampling machine and machine samples were taken as described above, except that alternate ('odd' and 'even') sample grabs were bulked separately to give two samples for comparison. The sampling scoop moved into the falling stream 12-16 times while 4 cwt. of flowers were passing over the belt, so that the 'odd' and 'even' samples were each made up of 6-8 separate grabs. The bulk of the material which was not sampled was collected in bins and this process was then usually repeated. The 4-cwt. batches can be regarded as 'sections' of the flow from much larger bulks, and any consistent biases found on the test sections must also occur when sampling larger consignments.

In experiment 6 the flowers were sampled once by M_1 and once by M_2, and in experiments 7 and 8, they were sampled twice by each machine. The machine M_2 could not be used subsequently (due to an explosion in the factory) and for experiments 9-13 the flowers were sampled three times by M_1. Handling of the flowers before using them for the reference sampling investigation probably somewhat increased the extent to which they were broken up, but did not result in much more mixing than that occurring incidentally when breaking up bales or emptying sacks of flowers. Each machine sample, which weighed about 2 lb. was ground in the Wiley mill and reduced (see Section 2c) to two 50 g. samples, one of which was retained at E.A.E.C. and the other sent to T.P.I. for analysis.

Table V gives the percentage pyrethrin content of the flowers used in each experiment, estimated from the reference samples obtained by riffling the bulk as described in Section 2e, and the average differences between these estimates and those from the machine samples, based on combined results from the two laboratories.

TABLE V

Estimated Biases in Pyrethrin Content of Machine Samples

Experiment	Pyrethrin content *per cent.* (R)	Difference between machine and reference sample estimates			Estimated percentage bias	
		M_1-R	M_2-R		M_1	M_2
6	1·50	+0·049	+0·020	±0·013	+3·3	+1·3
7	1·23	+0·006	−0·008		+0·5	−0·6
8	1·44	+0·010	+0·006		+0·7	+0·4
9	1·38	+0·014			+1·0	
10	1·40	+0·026		±0·010	+1·9	
11	1·11	+0·040			+3·6	
12	1·20	+0·024			+2·0	
13	1·29	+0·015			+1·2	

Clearly, the machine samples tended to have a slightly higher pyrethrin content than the reference samples and thus to overestimate the percentage pyrethrins in the bulk. Averaged over these experiments, the bias for M_1 was estimated to be $+0{\cdot}024 \pm 0{\cdot}005$ from the E.A.E.C. results and $+0{\cdot}002 \pm 0{\cdot}006$ from the T.P.I. results, or 1½-2 per cent. of the mean pyrethrin content.

The values of M_1-R for the individual experiments (Table V) were all positive; they average $+0{\cdot}023$ and have a standard deviation of $\pm 0{\cdot}015$. The smallest estimated biases were for experiments 7 and 8, for which unbaled flowers were used. As far as could be detected by sieve analysis (Table I) the flowers used for these experiments were no less fragmented than those from several of the bales used. The largest estimated biases were for experiments 6 and 11, which had respectively the highest and lowest pyrethrin contents of the series.

Thus, having due regard for the sampling errors of the estimates for the individual experiments, the results indicate that the bias of M_1 was fairly consistent over the range of conditions covered. Samples taken by this machine are subject to an upward bias of about 2 per cent. for pyrethrin content and there is no reason to expect a much greater bias, or for that matter a negative bias, under normal conditions. A bias of this order may not be a serious defect in practice as it is little greater than the E.A.E.C. laboratory error and routine determinations are normally based on one, or at most two, analyses.

The limited information obtained on the performance of M_2 suggests that any bias to which this sampling machine is subject is even smaller, but a further series of tests of M_2 against the reference sampling method or, perhaps more conveniently, of M_2 against M_1, would be advisable to check this.

As remarked in Section 3b one of the more obvious sources of bias in the samples taken by these machines is that they must sample a smaller proportion of that part of the stream from the conveyor belt which falls closest to the belt; this part of the stream will be referred to as the 'back'. In experiments on dried flowers from three different consignments, the back and front halves of the horizontal cross-section of the stream falling from the conveyor belt of M_1 were sampled separately. This was done by stopping the conveyor belt, sweeping it clean and then holding a divided polythene bag in position to collect the two halves of the cross-section separately when the machine was restarted and flowers fed by the hopper began to fall from the end of the belt. After about 10 seconds, the top of the bag was closed and the machine stopped. This process was repeated three times for each of the three consignments. The individual samples were ground, reduced as in Section 2c and analyzed; the results are summarized in Table VI. Flowers which had been baled were used for the second of these experiments, and unbaled flowers for the other two.

Between a third and a quarter of the total weight of flowers was in the back half of the cross-section of the falling stream. In all three experiments the percentage pyrethrin content in the back half of the stream was substantially less than that at the front. Thus, unless other factors intervene, samples taken by this type of machine must tend to give overestimates of pyrethrin content.

TABLE VI

Pyrethrin Content at Back and Front of the Stream falling from Conveyor belt (M_1)

Percentage of total weight in back half of stream	Estimated Pyrethrin Content *per cent.*				Estimated percentage bias if sampling time for back reduced by	
	Front (F)	Back (B)	Difference (F−B)	Whole Stream	One third	One half
26	1·29	0·86	+0·43 ±0·01*	1·18	+2·5	+4·0
36	1·13	0·95	+0·18 ±0·02	1·06	+1·4	+2·4
27	1·32	1·17	+0·15 ±0·02	1·28	+0·8	+1·3

* Standard errors of the average difference over the particular three segments sampled

From visual observation, and by studying a series of photographs, it was very approximately estimated that the time during which the sampling machine M_1 was collecting from the front half of the falling stream but not from the back was about one-third of the total sampling time. At the right-hand side of Table VI estimates are given of the percentage pyrethrin content of the whole cross-section of the falling stream for the three experiments, and of the percentage biases which would result if the sampling time for the back half of the stream were cut by a third or a half. These biases are of much the same order as those found when comparing M_1 samples with the reference samples (Table V). Thus the small bias found in practice can be accounted for numerically by the differential sampling of the falling stream as the sample scoop moves in and out.

This bias could probably be further reduced by increasing the total time during which the scoop remains in the sampling position, but this would increase the total size of sample taken, unless the interval between consecutive sample 'grabs' was also increased. Should it be considered of sufficient practical importance, this source of bias could be entirely eliminated by using a sampling machine with a scoop or bin which collected from the whole cross-section of the falling stream, moving transversely across the stream rather than up and down through it. With such a sampler, it would not be necessary for the collecting bin to move rapidly through the stream, as the size of the sample could be controlled to some extent by choosing an appropriate width for the bin in the direction of its motion. This type of machine would be preferable in principle to the machines at present in use at E.A.E.C. if it could be made equally reliable in operation.

In addition to estimating bias, experiments 6-13 gave information on the variability of pyrethrin content between different machine samples taken from the same batch of flowers. For M_1, the standard error of an estimate from an 'odd' or 'even' grab sample from 4 cwt., based on two E.A.E.C. and two T.P.I. laboratory determinations, was ±0·017 (32 d.f.) and none of the individual estimates differed from their mean by more than twice this standard error.

Allowing for laboratory errors, the estimated variance between the actual percentage pyrethrin contents of 'odd' or 'even' grab samples from 4 cwt. was 169×10^{-6}. Probably by chance, this is even less than the estimated variance for a riffle sample of comparable size (bottom line of Table II) and implies that the mechanical sampling method is very efficient. The variance for the total sample, 'odd' plus 'even' grabs from 4 cwt. of flowers, can be estimated as 84×10^{-6}.

When sampling from a consignment made up of several bales (or equivalent quantities) which may differ considerably in their percentage pyrethrin content, there will be a further contribution to the sampling variance, because the machine will not collect exactly the same weight of flowers from each bale. This contribution will be approximately

$$2p^2.\sigma^2/n$$

where p = the percentage standard deviation of the weight of flowers sampled per bale.

σ^2 = the variance of the percentage pyrethrin content between bales.

and n = the number of bales sampled.

With a fixed setting of the sampling machine the standard deviation of the weight sampled from a bale is less than 10 per cent. of the mean, so in practice this contribution will also be quite small.

As an example, suppose a sample is taken by M_1 from a 1-ton batch of flowers (5 bales), that this sample, weighing about 20 lb., is then reduced to 50 g. of ground flowers as described in Section 2f, and that two spectrophotometric determinations are then made in the E.A.E.C. laboratories. Taking $p = 0{\cdot}1$ and $\sigma = \pm 0{\cdot}2$ (a higher value than is likely in practice), the variance of the estimated percentage pyrethrin content of the bulk will be made up approximately as follows:

Primary sampling by machine		Sample reduction	Laboratory
Within bales	Between bales	by riffling	error
$\frac{1}{5} \times 84 \times 10^{-6}$ +	$\frac{1}{5} \times 800 \times 10^{-6}$ +	300×10^{-6} +	$\frac{1}{2} \times 300 \times 10^{-6}$
i.e. 17×10^{-6} +	160×10^{-6} +	300×10^{-6} +	150×10^{-6}

$$= 627 \times 10^{-6}$$

The standard error of the estimate will therefore be $\pm 0{\cdot}025$ and, of the total variance, machine sampling will be responsible for about 28 per cent., sample reduction for 48 per cent. and laboratory error for 24 per cent. This is an extreme example, and in sampling from large bulks, or from bales which do not differ greatly in pyrethrin content, one can readily calculate that the proportion of the total variance which is due to machine sampling will be much smaller.* Thus the contribution of primary machine sampling to the random errors of estimation will not be important in practice.

3d. *An alternative method of sampling baled flowers*

An alternative method which has been used for sampling baled

* E.g., the variance due to primary machine sampling from a consignment of 25 tons would be approximately $\frac{1}{25}(17+160) \times 10^{-6} = 7 \times 10^{-6}$ which is trivial.

pyrethrum flowers is to take sample cores from the unbroken bales, using a steel tool with an inside diameter of 2 in. which cuts to a depth of 7 in., giving individual samples of about 5 oz. of flowers.

Using a tool of this type, cores were taken from the bales of flowers used in experiments 10-13 before they were broken up. Eight cores were taken, one from each quarter of the two opposed compressed faces of each bale. The individual samples were ground, reduced as in Section 2c, and two pyrethrin determinations were made on each, half of the samples being analyzed at E.A.E.C. and half at T.P.I.

The estimated standard deviations of the pyrethrin contents of the individual core samples were as follows:

Experiment	10	11	12	13
Standard deviation	±0·023	±0·118	±0·091	±0·012

Evidently the bales used for experiments 11 and 12 were more heterogeneous than the other two bales, and the results suggest that one cannot rely on obtaining estimates of required accuracy by taking a specified number of core samples from a bale. Even on the basis of the average variance for these four experiments, it would be necessary to take about 16 cores to obtain an estimate as accurate as that from a single 2-4 lb. riffle sample.

Satisfactory estimates of the pyrethrin content of a consignment might be obtained by taking core samples from several bales, but the accuracy attained would depend on the variability of pyrethrin content between bales. In one test, individual cores from 135 bales of a consignment were bulked in 15 groups of nine cores before sample reduction and analysis. The standard deviation of the estimated mean pyrethrin content based on a single bulked sample was ±0·026.

Due to the large sampling errors for individual cores, further work would be necessary to decide whether core sampling is biased. The possibility of bias cannot be ruled out, particularly as the bales are almost 3 ft. thick and flowers at the centre cannot contribute to the samples.

4. *Deterioration of ground pyrethrum flowers*

During the investigation, repeat laboratory determinations were made on a large number of samples. Often these determinations were made within a few days of each other, but sometimes the time interval was longer and some information was therefore obtained incidentally on the rate at which the pyrethrin content of the finely ground samples declined. Table VII gives a number of estimates of the average loss in pyrethrin content per week for samples kept in tins at room temperature in the two laboratories.

The E.A.E.C. figures relate to determinations made during February-March, the first determination being made within a day or two of grinding the flowers and the second determination 2-7 weeks later. The T.P.I. figures cover subsequent periods of about three months during March-June 1960.

The estimated average loss at E.A.E.C. was 0·017, or 1-1½ per cent. of the total pyrethrin content per week. At T.P.I. during the subsequent

TABLE VII

Estimated losses in Pyrethrin Content per week for Ground Flowers

E.A.E.C.		T.P.I.	
Weeks between determinations	Estimated loss per week	Weeks between determinations	Estimated loss per week
2	−0·028	11	0·000
2	−0·022	11	−0·001
3	−0·020	11	−0·006
3	−0·006 ±0·004	11	−0·006 ±0·002
3	−0·022	12	−0·006
4	−0·013	12	−0·003
5	−0·010	14	−0·011
7	−0·012	14	−0·007
	−0·017		−0·005

period the estimated loss was 0·005, or less than ½ per cent. of the pyrethrin content, per week. Although the conditions under which the samples were kept in the two laboratories were not identical, it seems likely that the rate of loss was higher soon after the samples were ground than subsequently, and differences between the individual E.A.E.C. estimates of loss are also consistent with this.

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Grading of Copra: Tongan Samples Examined

By P. Beevor and W. D. Raymond

During a visit to the T.P.I. laboratories, H.R.H. Prince Tungi of Tonga asked that samples of Tongan copra should be examined. These samples were submitted by the Department of Agriculture in Tonga in July 1959. Their examination gave the following results.

Description of Samples

The following samples were received:

Grade H.A.D.1, net weight 24 lb. 8 oz.
Grade H.A.D.2, net weight 28 lb.
Grade F.M.S., net weight 2 lb. 8 oz.
Grade Reject, net weight 33 lb. 8 oz.

Examination

The samples were examined visually according to the grading table attached to this report, with the results set out in Table I.

Moisture (loss at 105°C.) and oil contents were determined by the method described for oleaginous seeds in I.U.P.A.C. Standard Methods for the Analysis of Oils and Fats, 1954. The free fatty acid content of the extracted oil was determined according to the method described in British Standard, 684:1958. The results of this examination are given in Table II.

A number of pieces showing insect damage selected from each sample was investigated at the Pest Infestation Laboratory and the results are given in Table III.

Table I

Results of Visual Examination by Grading Table

	H.A.D.1	H.A.D.2	F.M.S.	Reject
Average Colour . .	4	4	4	1
Cleanliness and Smell .	3	3	3	1
Size and Shape . .	4	3	3	2
Condition . . .	4	3	3	2
Dryness . . .	4	3	4	3
Assessment (Max. 100) .	95	80	85	45

TABLE II

Results of Examination

	H.A.D.1	H.A.D.2	F.M.S.	Reject	Usual Range
Volatile matter of copra (loss of weight at 105°C.) per cent. . . .	4·4	4·9	3·8	4·2	4–7*
Oil content (moisture-free basis), per cent. . .	66·8	65·8	64·7	67·1	63–68*
Free fatty acid of oil (calculated as oleic acid), per cent.	1·1	1·2	1·0	4·5	Not more than 5·0†

* E. W. Eckey, *Vegetable Fats and Oils*, Reinhold Publishing Corp., New York, 1954, p. 311.

† British Standard 628:1950 for Crude Coconut Oil.

TABLE III

Results of investigation of sub-samples by the Pest Infestation Laboratory

Grade H.A.D.1 .	Moisture content about 4·0 per cent. (by copra probe). Infested with *Oryzaephilus mercator*. Somewhat rubbery, suggesting too quick drying.
Grade H.A.D.2 .	Moisture content about 4·0 per cent. (by copra probe). Infested with *Oryzaephilus mercator*, *Necrobia rufipes* and *Ephestia cautella*.
Grade F.M.S. . .	Moisture content about 4·0 per cent. (by copra probe). Infested with same species as Grade H.A.D.2, but more heavily so.
Grade REJECT .	Moisture content about 4·0 per cent. (by copra probe). Heavily infested with *Oryzaephilus mercator* and *Necrobia rufipes*, the cause of the severe 'pitting' observed.

TRADE VALUATION

The samples were also submitted to a firm of oil seed crushers who are experienced in handling copra. They reported as follows:

'The analyses of the samples of copra submitted are as follows:

	Oil *per cent.*	F.F.A. *per cent.*	Moisture *per cent.*
Grade H.A.D.1 . .	65·6	0·91	3·8
Grade H.A.D.2 . .	65·0	1·52	3·9
Grade F.M.S. . .	66·5	0·90	3·0
Grade REJECT . .	66·8	3·70	4·7

As you will see, the F.F.A. was reasonably in line with the graded quality.'

The above report is particularly interesting in that it shows a greater proportion of oil present in the reject grade of copra than in the other grades. This is a phenomenon which is frequently observed with commercial consignments and since the value of any consignment is predominantly affected by the proportion of oil that the material contains, reject grades of copra may command prices not much below those for the highest quality material. It should, however, not be concluded by the primary producers that quality is of no importance economically. The

apparent anomaly is due to the loss of weight caused by the attacks of moulds and insects, some of which were found and reported in Table III of the report. Moulds and insects may preferentially consume the protein rather than the fat present in the copra and although the quality of the fat invariably deteriorates due to lipolysis the actual percentage of fat found on analysis in the partly consumed material may be greater than it was in the original. There has, however, been a considerable loss in weight caused by the depredations of the insects and moulds and hence the total weight of material available for sale is considerably less than it would have been if a higher grade of product had been produced initially.

The grading of copra has been under close study by the main firms concerned with crushing this material in Europe and reports have been made from time to time to the Congress of the International Association of Seed Crushers. The general tendency in trade circles is to purchase copra on the basis of its fat content with a discount for unsatisfactory colour or high fat acidity. This may again appear anomalous because it disregards the question of moisture, but since the oilseed crusher is solely concerned with the yield of oil from a given weight of copra, moisture is not of prime interest to him. It is, however, of some concern to the primary producer and is undoubtedly the most important factor causing copra to deteriorate. This has been clearly recognised by the Working Party on Copra Quality and Grading set up by the Food and Agriculture Organization. Unless the moisture content of the copra is reduced to a safe level as soon as possible after opening the nuts, attack by wild yeasts soon occurs and this attack is quickly followed by attack from bacteria, followed often by the production of alcohol, inviting the prompt attention of insects. The rapid attack by insects is followed by further mould attack, usually giving rise to a red or brown colouration of the tissues, and by further depredation by insects. Perhaps the most harmful of the various biological agencies attacking copra are the mites which break the copra down into dust.

For the purpose of the first stages of rural trading moisture should be not more than 9 per cent. and it is advantageous that all areas producing copra should adopt some system whereby the moisture content of the copra is controlled. Often the best way of controlling moisture is by the rough and ready methods of visual examination such as those described in the grading table or by F. C. Cooke.[1] While such visual systems are extremely valuable in the grading of copra they can, with advantage, be supplemented by the use of a meter which can be simply and convincingly demonstrated. There are various meters on the market but most of them require rather elaborate preparation of the material before it can be tested. In a recent meeting at the Tropical Products Institute attended by members of trade bodies in the United Kingdom, agricultural officers serving overseas, and others, it was concluded that the Pest Infestation Laboratory meter at present under development, which determines fairly accurately the water content of copra between 4 and 10 per cent., is eminently suitable for field use. In collaboration with the Pest Infestation Laboratory the Institute have made about half a dozen

of these instruments available for field trial in selected Commonwealth countries. It is hoped to receive reports from these countries in due course.

Reference

(1) Cooke, F. C., 'Practical Aspects of Copra Deterioration', *Gen. Ser. Dep. Agric. S.S. or F.M.S.*, 1937, No. 28, 41-5.

Copra Grading Table

Average Colour		Cleanliness and Smell		Size and Shape		Condition: Decomposition, Dust and other Extraneous Matter		Dryness		
4	White	4	Clean Sweet	4	Large (not smaller than 6 pieces per nut) Thick	4	None	4	Pearly lustre Biscuit hardness	Not exceeding 4 per cent. moisture
3	Grey to Pale Brown	3	Slightly dirty ,, rancid ,, mouldy ,, smoked	3	Medium with up to 5 per cent. of small pieces	3	Up to ¼ per cent. Copra dust through ⅛″ screen Up to 5 per cent. slightly decomposed pieces Slight weevil attack	3	Breaks with dull snap Dark pearly lustre	4-5 per cent.
2	Brown	2	Dirty; Rancid; Mouldy: Smoked	2	Small with up to 5 per cent. thin pieces	2	Up to ½ per cent. Copra dust and dirt Up to 10 per cent. slightly decomposed pieces	2	Bends on breaking Faint water line	5-7 per cent.
1	Dark Brown	1	Very dirty ,, mouldy ,, smoked	1	Very small ,, thin	1	Up to 1 per cent. dust and dirt Up to 30 per cent. partially decomposed pieces Rubbery; Immature	1	Soft, rubbery. Distinct water line Breaks with dull thud	7-8 per cent.
0	Reddish Black to Black	0	Rotten Charred to burnt	0	Shapeless Distorted	0	Sand, stones, coral, irons, and other metals. Badly decomposed Very rubbery—crinkled	0	Soft, spongy Difficult to break and no snap	8-9 per cent.

Improve the quality of your Copra by the use of this chart: half-marks may be used where deemed necessary.

Plantation to F.M.S. Grade . . . 60–100 marks.
,, ,, F.M. ,, . . . 45–59 ,,
Reject for export . . . . . Under 45 marks.

The grade is determined as follows:

Example.—Colour 2; Cleanliness and Smell 3; Size and Shape 2; Condition: Dust, etc. 3; Dryness 2; Total $12 \times 5 = 60$ marks out of a possible 100.

Notes

Cacao Beans from Tanganyika.—Two samples of cacao beans were forwarded to the Tropical Products Institute by the Field Officer, Tanga and Pangani Districts, Department of Agriculture, Tanganyika Territory.

The beans had been grown in the Tanga District of Tanganyika Territory. Information on the commercial value of the beans was requested.

Description

*Sample A*1 consisted of 1 lb. of cacao beans which had been fermented for 72 hours, washed in water and then sun-dried. The beans showed some evidence of insect attack.

*Sample A*2 consisted of 1 lb. of cacao beans which had been fermented for 96 hours, washed in water and then had alternate days of shade and sun drying. The beans showed no evidence of insect attack.

Commercial Valuation

Representative portions of the samples were submitted to two firms of cocoa and chocolate manufacturers for their opinions on the quality and commercial value of the beans. The firms reported as follows:

Firm A—

'*Sample A*1. The beans in this sample were of good size, 200 weighing 227 grams, and they were predominantly Criollo but poorly fermented. The shell percentage was 8·6 per cent. but it was very brittle. Chocolate made from these beans was astringent and unpleasant.

*Sample A*2. 200 beans weighed 280 grams with the percentage of Criollo much less than in Sample A1. The non-Criollo beans included a number of purples, leaving room for improvement in fermentation. The shell content was 7·3 per cent., the shell again being very brittle. Chocolate made from this sample showed a slight improvement over that made from Sample A1.

Our general comments are as follows:

The samples contained a mixture of Criollo and Forastero beans and a mixture such as this inevitably gives rise to fermenting difficulties because of the differing requirements for the two types.

The washing which the samples underwent, followed by the drying, probably gave rise to the brittle nature of the shell. In bulk supplies this would be a disadvantage not only because of the increased amount of infestation—broken shells obviously enable insects to get at the nib—but would involve a large commercial user in considerable difficulty in processing.

You probably know that the bulk of the cocoa used in this industry is of the Forastero type as this has been found the most suitable for the production of milk chocolate. Criollo beans amount to between 5 per cent. and 10 per cent. of the total world production and although at times they command a premium, this is not always the case.'

Firm B—

'*Sample A*1. The beans were clean but rather brittle and had suffered considerable breakage. Their size was on the small side (weighing 103·6 g. per 100 beans) but they were plump. The cotyledons were mostly of the white type or with a tinge of purple colour, and appeared to have received only a very slight fermentation, most of the cotyledons showing either the white of unfermented beans of this type or showing only a very slight degree of fermentation. Clearly these beans are from almost pure Criollo type trees.

The flavour of the chocolate made from these beans is so astringent that their intrinsic value cannot be assessed, but such flavour as could be detected behind the astringency was earthy or mouldy. The colour of the chocolate is very light indeed, like a milk chocolate.

Obviously, the fermentation of this sample is quite unsatisfactory, the beans being useless for chocolate making, and no assessment of their value is possible. Doubtless some fat could be extracted from them, but until their fermentation is improved they are of no interest to a chocolate manufacturer.

Sample A2. The beans were clean but rather brittle and had suffered considerable breakage. Their size was large (100 beans weighing 139 g.) and they tended to be flat in shape, and it would be expected that the trees were genetically different from those giving Sample A1. The colour of the cotyledons varied from white to purple types and showed considerably more fermentation than Sample A1. The white types had mostly been properly fermented to a cinnamon colour, but the darker beans showed a good deal of purple. It appeared that fermentation was to a somewhat lower degree than the optimum. The beans appear to be derived from a mixture of Criollo and Forastero types and include many hybrid beans.

The flavour of the chocolate made from this sample is not astringent but is unduly bitter. There is a mild type of cocoa flavour without much depth or strong aroma, but it is a clean flavour which could be useful for certain purposes.

The colour of the chocolate is somewhat lighter than normal.

It is probable that the fermentation of this sample could be improved so as to reduce the bitterness and perhaps to enhance the cocoa flavour. There is some demand for these milder and lighter coloured cocoas, and it is quite possible that such a cocoa might, in relatively small quantities, be able to command a small premium over West African cocoa, and especially if the bitterness could be reduced.

In general, it is doubtful whether the procedure of washing the beans in water after fermentation is advisable. This is not the practice in the majority of the major cocoa growing areas, including those which produce the better quality cocoas, and is probably the main reason for the undue breakage shown by these beans, a serious practical drawback because of the difficulty of securing even roasting and the increased liability to insect infestation. Indeed, Sample A1 contained a half grown *Ephestia* larva.

Another possible effect of washing is to reduce the strength of flavour developed. In view of the greater Criollo content of beans of Sample A1 it is surprising that its three-day fermentation has given such a poor result compared with four days for the hybrid cocoa of Sample A2. Perhaps, because of the small quantity of beans, the fermentation did not develop its proper activity (temperature in the beans is a coarse measure of activity), and the slower drying of Sample A2, resulting from the alternate days of shade and sun drying may have allowed more fermentation activity during the first days of drying. While such alternation is not a recognised procedure as a rigid process, it is certainly true that many producers of quality cocoa do consciously restrict the rate of drying according to their judgment of the amount of fermentation which has taken place in the sweat box.

With regard to the market value of these cocoas, the beans in Sample A1 are of no value for chocolate manufacture. In the case of Sample A2 the beans might possibly be of some interest on grounds of flavour. Small quantities might attract some premium over West African cocoa.'

Comments

From these reports it can be seen that Sample A2 is superior in quality to Sample A1, but the quality of both the samples could be considerably improved. Both samples consist of a mixture of Criollo and Forastero types of cacao beans, the beans in Sample A1, however, being predominantly of the Criollo type. Though Sample A1 is not recommended for making chocolate, this type may find other uses.

The beans, in each case, are rather brittle and their flavour unsatisfactory. The brittleness of the beans is probably due to the fact that the beans were washed in water after they had been fermented; this is not the usual practice, and it is suggested that this step should be left out of the processing. Washing may also tend to affect the flavour of the beans adversely.

Flavour is also affected by the fermentation process. Criollo beans are less

astringent than the Forastero type and require less time for complete fermentation. The astringency of Criollo cacao is normally reduced to a satisfactory level after fermentation for two or three days, whereas Forastero beans require a fermentation period of five to seven days. Owing to the different times of fermentation for each type of bean, it is difficult to ferment samples of mixed beans to give a satisfactory product, and it is recommended that the beans should be sorted into two groups before fermenting and mixed again afterwards if desired.

W.D.R., P.K.C.

Book Reviews

BOTANY, AN INTRODUCTION TO PLANT SCIENCE, by W. W. Robbins, T. E. Weier, and C. R. Stocking. Second Edition, 1957, ix + 578 pp. (New York and London: John Wiley & Sons Inc., London: Chapman & Hall Ltd.) Price 56*s*.

This book is a revised edition of a textbook on botany intended for students with little previous scientific training. The style of presentation is pleasant and the method of approached should stimulate interest. As in the first edition the more difficult aspects of the subject are introduced by first describing known reference points such as geographical and agricultural topics, and the morphology and physiology of the angiosperms are described before the student is introduced to the more primitive plants. Many small changes have been made in this edition and the chapters on physiology have been completely re-arranged; the functions of the plant as a whole now being described instead of discussing the functions of the parts separately under the sections on their morphology. The chapter on viruses could have been rewritten to give greater emphasis to plant viruses and to give some indication of the recent work on virus structure and composition.

The illustrations are numerous and generally good though some do not merit inclusion as they add nothing to the descriptions in the text.

This book is intended for North American students and some of the examples may be unfamiliar in Europe but it would nevertheless be a useful textbook for sixth form or first year university students.

B.R.S.

THE CHEMISTRY AND TECHNOLOGY OF CEREALS AS FOOD AND FEED. Edited by Samuel A. Matz. xv and 732 pp., 9 × 6. (The Avi Publishing Company, Inc., Westport, Connecticut, 1959.) Price $16.

Until now there has been no single book which dealt with all the cereals and with the industries based upon them. Indeed for some cereals (e.g. rye, sorghum) and some industries it has been difficult to find even specialized works to which the student could refer. This deficiency has reflected the rather wide range of knowledge and expertise which is needed to cover the subject adequately. Hence it is natural that a book which fills the gap so effectively as this must be a compilation to which a considerable number of specialists have contributed, and it is natural that the resulting volume should have the merits and defects of all such books.

The defects, however, are less than in many such works which have appeared in recent years. Each article has been written to conform generally to a common editorial plan and considerable efforts have clearly been made to secure a degree of uniformity in style and balance between the authors.

The articles are grouped under three headings. In Section I, one article deals with each of the seven major cereals, wheat, corn (maize), oats, barley, rye, sorghum and rice. An eighth article deals briefly with millet (mainly *Panicum miliaceum*), wild rice, rice grass, and adlay ('Job's tears'). In Section II, various processing methods are dealt with in twelve chapters. Six of these deal with what might be called primary processes, using cereals as their raw materials, namely, milling (wheat, corn, oats, rye and sorghum), wet milling, feed manufacture, production of oils from cereal grains, rice processing and malting. The remaining six deal with commercial baking, macaroni production, manufacture of prepared mixes, manufacture of special dietary foods, manufacture of breakfast cereals and brewing. Section III contains five articles on matters of general interest concerning cereals and cereal products. These are: cereal starches, flavour staling resulting from lipid deterioration; processed cereals in human nutrition; supplementation of cereal-based animal feeds, and insect and rodent infestation of cereals.

The avowed aim of the book is to give width rather than depth of coverage but a useful bibliography is appended to each chapter which may enable the student

to extend his reading usefully on each subject. Nevertheless, for many users it would have been better if the chapters on the secondary cereal-based industries, which are generally well documented elsewhere, had been omitted, so that more attention could have been given to general cereal technology, especially in grading, storage, and handling. The editor admits that emphasis has been placed upon procedures which are usual in the United States, as is inevitable with an all-American authorship, but this does not detract in any way from the usefulness of the very great amount of basic information which is given. Generally, differences between U.S. and European practice are noted in the processing chapters and a number of the illustrations in the article on milling show equipment of European types.

The articles on the cereals follow a common plan. Each begins with a short account of the present status of the crop and reviews its known history. The taxonomic position of the cereal is given and usually a certain amount of other related botanical information. This is followed by a section on cultural methods which varies greatly in the different chapters, being especially long in the case of rice. Standards and quality are next dealt with, details usually being given of the relevant U.S. grain standards. Some data on production statistics are given for most of the grains and in every article a short section deals with drying and storage. The latter sections, naturally, tend to be very similar in each article and in later editions it might be more useful to deal with these subjects more thoroughly in a general article in Section III.

The book is well produced and has a short, but adequate, index.

T.A.O.

PERFUMES, COSMETICS AND SOAPS, with Special Reference to Synthetics. By William A. Poucher, F.P.S., F.R.P.S. Volume Three, being a Treatise on Modern Cosmetics. Seventh Edition Revised 1959. Pp. xi + 260, $8\frac{1}{2} \times 5\frac{1}{2}$. (London: Chapman & Hall, Ltd.) Price 45*s*.

As the author indicates in his preface to this new edition, the cosmetic industry has expanded enormously during the past decade. The purchasing public has become more discriminating, and cosmetic chemists have been compelled to pay greater attention to research and manufacturing efficiency. Their work has been facilitated by the vast background of investigation initiated and maintained by makers of raw materials; this has resulted in the evolution of numerous series of specialities, many of which, however, are known only under pseudonyms and of which their chemistry is obscure. Nevertheless, the author says, the fundamental background of the industry has not changed materially.

Not unnaturally, the new edition reflects the opinion expressed by the author, for it closely resembles the previous edition; however, new material includes references to two non-proprietary chemicals of known chemical composition which have become important raw materials in the formulation of toilet preparations, namely, isopropyl myristate and hexachlorophene.

Isopropyl myristate is a colourless, almost odourless liquid, miscible with oils, fats, waxes and fatty alcohols, but insoluble in water, glycerol, sorbitol and carbitol. It has the effect of reducing the 'greasiness' of water-in-oil emulsions in which it is used, a property of considerable value in cosmetics of this type; when mixed with most waxes, particularly those of micro-crystalline structure, it yields a thixotropic gel. This property has been utilized in lipstick formulation and has facilitated the production of the 'creamy', glossy types now fashionable. Type formulae, illustrating the uses of isopropyl myristate in lipsticks and other preparations, are provided.

Hexachlorophene is a bacteriostatic and bactericide which differs from other antiseptics in retaining its activity in the presence of soap. It was developed primarily to meet the need for a truly antiseptic soap, but it is now an accepted constituent of many cosmetics as well. Examples of its use in talcum powders, after-shave lotions and perspiration deodorants are given.

The paragraph on lipstick colours has been revised. It is, however, very brief and more detailed information about these colours, including some reference to United States legislation on the subject, would not have been out of place. Other

modern developments which have found a place in this edition are the cold hair-waving preparations, based on salts of thioglycollic acid, and pre-electric shave lotions.

The text contains a very few errors, of minor character, but which, unfortunately, as in the companion volumes, have been allowed to persist from earlier editions. The book is well presented but the new binding is less pleasing than the familiar blue and gold flexible covers of the earlier editions. There is no reason to doubt, however, that the new edition will continue to enjoy the success accorded to its predecessors.

E.B.

PLANTS AND ENVIRONMENT. A TEXTBOOK OF PLANT AUTECOLOGY, by R. F. Daubenmire. Second Edition, x + 422 pp., 92 figs., 9 × 6. (New York: John Wiley & Sons, Inc.; London: Chapman & Hall Ltd., 1959.) Price 56*s*.

The factors affecting plant growth and development are dealt with under the headings of soil, water, temperature, light, atmosphere, fire, biotic, the environmental complex, ecological adaptation and evolution. Methods are described for the practical determination of a number of conditions. The illustration of conditions, both by written examples and by photographs is largely but not exclusively related to the North American continent.

The photographic illustrations are of mediocre quality and it is unfortunate that the opportunity was not taken to make improvements in this respect in the second edition.

This second edition, while retaining the form of the original which was published in 1947, has been extended to include the results of scientific research in the field of plant ecology during the past eleven years. The list of references has likewise been expanded and now numbers 773 citations.

M.O.

BANANAS. By N. W. Simmonds. xvi + 466 pp., 8¾ × 5¾. (London: Longmans, Green & Co. Ltd., 1959.) Price 45*s*. net.

The appearance of a comprehensive review of the literature of bananas is especially welcome since for too long there has been no up-to-date work in this field. Moreover, it has been written by an author who has worked for many years at the Imperial College of Tropical Agriculture, Trinidad, where he is Senior Cytogeneticist, Regional Research Centre, in charge of banana breeding, and who is widely recognized as a leading authority on his subject.

The book, like other publications in the Tropical Agriculture series, is designed to meet the needs of three groups of readers: research workers, government agricultural officials and the planters. Two opening chapters describe the botany of edible bananas and these are followed by a section of three chapters devoted to the classification of banana cultivars. The next section deals with crop production factors, i.e. climates and soils, planting and management, and harvest, transport and ripening, while later chapters deal with composition and utilization, production and economics, the history of bananas and the banana trade, and, the author's own particular subject, banana breeding. In view of Professor Wardlaw's book on diseases of the banana, which, although published in 1935, has not been substantially outdated, Mr. Simmonds has limited his treatment of diseases to a chapter providing fairly detailed accounts of the three major diseases, Panama or banana wilt, leaf spot or Sigatoka, and bunchy top, together with brief references to recent literature on those of lesser importance. As regards banana pests which are relatively unimportant and concerning which no monograph exists, condensed accounts of the five most important pests, with an annotated list of others, have been provided in a companion chapter.

The author's account of the utilization of bananas serves to emphasize the negligible importance of processed banana products in international trade, a factor which is a considerable handicap to the industry. A minor point is that banana figs are described as slivers or slices whereas, for the very limited United

Kingdom import trade, the bananas are preferred dried whole although the ends should be removed. An interesting development to which, unfortunately, no reference is made is the production of canned banana puree in the Dominican Republic, mainly for use by manufacturing trades in the United States (*Food Technology*, 1957, **29**, No. 4, 66).

The book is extremely well illustrated; a useful feature is that each chapter is provided with a summary and an excellent bibliography. Indeed it can be recommended to all concerned with this important fruit crop.

E.H.G.S.

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Compiled by Miss V. Clarke

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